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This document consists of _____ pages
No. 2 of _____ copies, Series B

ASD-TDR-62-1025
VOLUME I of V

(Unclassified Title)

FINAL REPORT FOR HIGH ENERGY FUELS PROJECT
VOLUME I. PRELIMINARY DESIGN FOR A LARGE SCALE BORANE PLANT

TECHNICAL DOCUMENTARY REPORT NR. ASD-TDR-62-1025. VOLUME I

JUNE 1962

CHEMICAL ENGINEERING BRANCH
MANUFACTURING TECHNOLOGY LABORATORY
AERONAUTICAL SYSTEMS DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

ASD PROJECT NR. 7-558b

DOWNGRADED AT 3 YEAR INTERVALS
DECLASSIFIED AFTER 12 YEARS
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AFN

Prepared Under Contract AF 33(600)-35745

AFN, Inc.
LOS ANGELES 5, CALIFORNIA
Author: Dr. T. W. Clapper

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June 1962

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FOREWORD

Volume One of this Final Technical Documentary Report presents a preliminary design for a large scale borane plant in fulfillment of Contract AF 33(600)-35745. The manuscript was released by the author on 15 June 1962 for publication as an ASD Technical Documentary Report.

This contract with AFN, Inc. comprised of American Potash & Chemical Corporation, FMC Corporation, and National Distillers and Chemical Corporation, with main offices located in Los Angeles, California, was initiated under Manufacturing Methods Project 7-558b, "High Energy Fuels Project". It was accomplished under the technical direction of Mr. Charles Tanis, Chemical Engineering Branch, (ASRCTC), Manufacturing Technology Laboratory, Aeronautical Systems Division, Wright Patterson Air Force Base, Ohio.

Dr. T. W. Clapper of American Potash & Chemical Corporation was Project Manager. Others who cooperated in the supervision of the work covered by Volume One were R. C. Rhees and C. B. Armstrong of American Potash & Chemical Corporation.

This project has been carried out as part of the Air Force Manufacturing Methods Program. The primary objective of the Air Force Manufacturing Methods Program is to develop on a timely basis manufacturing processes, techniques and equipment for use in economical production of USAF materials and components. This program encompasses the following technical areas:

Rolled Sheet, Forgings, Extrusions, Castings, Fiber and Powder Metallurgy
Component Fabrication, Joining, Forming, Materials Removal
Fuels, Lubricants, Ceramics, Graphites, Non-Metallic Structural Materials
Solid State Devices, Passive Devices, Thermionic Devices

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production programs. Suggestions concerning additional Manufacturing Methods development required on this or other subjects will be appreciated.

Volumes I, III, IV and V of this report are classified CONFIDENTIAL because they disclose new, novel and unique methods of producing and processing certain boron hydrides.

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VOLUME I - TECHNICAL DOCUMENTARY ABSTRACT REPORT

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The preliminary design of versatile 9.5 ton per day (nominal) high energy fuels plant incorporates new processes demonstrated on the prepilot and pilot plant levels. The simultaneous or virtually exclusive production of diborane, pentaborane, decaborane or HEF-3 is possible utilizing a direct, economical route and proven, refinery-like procedures.

Boron trichloride is directly reduced with hydrogen to form dichloroborane in a silver-lined, catalytic reactor operating at 1275°F. and 250 psig. The dichloroborane is separated, concentrated and disproportionated to form diborane in conventional absorption and distillation operations. The diborane is recovered and stored as a refrigerated (-20°F.) liquid in 99 per cent minimum purity and 95 per cent yield. High purity anhydrous hydrogen chloride is obtained as a by-product which can be reused for the manufacture of boron trichloride feedstock. Twelve tons per day of diborane are produced.

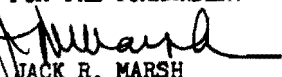
Diborane is pyrolyzed to form pentaborane and/or decaborane in a mild steel reactor at 575°F. and 75 psig. The reactor gases contain diborane and hydrogen in the mole ratio of 1 to 7 and are quenched after 2 seconds exposure to the high temperature in an inert solvent carrier, cyclohexane. The desired product(s) is separated, again with conventional stripping and distillation techniques, and lower molecular weight boron hydrides are returned to the reactor for conversion to the desired end product. The small amount of yellow solids filtered out as an undesired by-product may be processed to make boron trichloride, and the hydrogen released from pyrolysis may be purified and returned as raw material to the diborane plant. When decaborane alone is the desired product, 9.2 tons per day (87.2 per cent yield on diborane) may be produced either as a crystalline material of 99 per cent purity or as a 20 mole per cent cyclohexane solution for alkylation to HEF-3. When pentaborane is the desired product, 9.5 tons per day (87.4 per cent yield on diborane) are produced as a 99 per cent minimum pure liquid along with 0.56 tons per day of decaborane.

HEF-3 is produced by the continuous alkylation of decaborane in cyclohexane solution at 150°F. and 0 psig. Ethyl chloride is the alkylating agent and aluminum chloride is the catalyst. The proportion of di-, tri-, and higher alkylated decaboranes formed is controlled by limiting to 45 per cent the per pass conversion of decaborane to alkylated product. Subsequent distillation operations, carried out under reduced pressures to minimize thermal breakdown of the product, separate cyclohexane and decaborane for recycle as well as 9.4 tons per day (98.5 per cent yield on decaborane) of a high purity ethyl decaborane product.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:


JACK R. MARSH
Assistant Chief
Manufacturing Technology Laboratory
Directorate of Materials & Processes

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INTRODUCTION

Compounds of boron and hydrogen, as well as certain derivatives, possess unique properties which make them attractive as high energy fuels. These compounds, while similar in some respects to the hydrocarbons, have an approximately 50 per cent higher heat of combustion. For this reason they were originally considered as replacements for such hydrocarbon fuels as JP-4 in weapon systems employing air breathing engines. More recently, their use as rocket fuels has been given important consideration because of the high specific impulse developed by certain borane-oxidizer combinations. The wide range of physical and chemical properties characteristic of the stable boranes and their derivatives make them potentially suitable for a variety of applications.

Prompted by the potential importance of the borane compounds, AFN, Inc., at its own expense, constructed a pilot plant for decaborane production at Henderson, Nevada. Early, in 1957, high quality decaborane was delivered to the Navy and to various commercial and institutional laboratories. This activity demonstrated the specialized knowledge in borane chemistry which AFN, Inc. acquired through normal commercial enterprises, company sponsored research programs, and laboratory work performed under Air Force Contract 33(616)-448, 1956 Extension.

With this background, AFN, Inc. contracted [AF 33(600)-35745] in June 1957 to design, build, and operate a five pound per day process development plant for the manufacture of an alkyl borane, monoethyl decaborane. In the following four years, seven supplemental agreements to the original contract were issued. This development activity culminated in the design, construction and operation of a fifty pound per day pentaborane plant at Henderson.

Volume One of the Final Technical Documentary Report presents a preliminary design for a large scale borane plant and is in essence a summation of the knowledge gained over the entire program. Detailed information concerning the various developmental phases as they were carried out can be found in the other four volumes; it is this information which provided the basis for the final plant design.

A number of processes are essential to a fully integrated borane plant such as hydrogen and boron trichloride manufacture, and recycle of by-product hydrogen chloride. These operations were not under study in this project; therefore only general requirements are expressed in this report. Detailed information is presented for the three major steps involving boranes:

- Step I - Diborane Production
- Step II - Pyrolysis of Diborane to Produce
Decaborane (or Pentaborane-9)
- Step III - Alkylation of Decaborane

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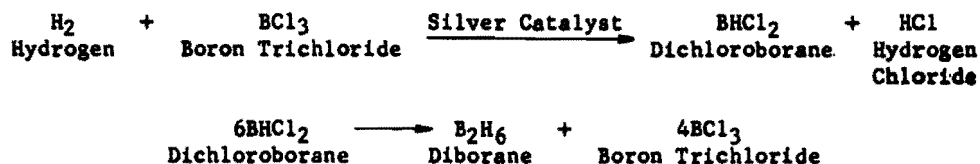
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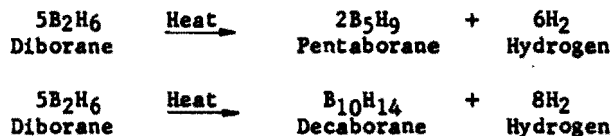
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While the initial APN process for making diborane involved the preparation of sodium borohydride from metallic sodium, the presently recommended method as demonstrated in the pilot plant utilizes the more direct and economical process of the catalytic reduction of boron trichloride with hydrogen. Chemically, the process involves only two reactions:



Side reactions are minor and can be controlled to produce high purity diborane in excellent yields.

Diborane, when pyrolyzed, forms a mixture of higher boron hydrides. The reactions can be controlled to provide a reactor effluent rich in the desired boron hydride, pentaborane-9 or decaborane. While a series of complex reactions are involved in diborane pyrolysis, the desired net reactions are:



A petroleum refinery type of operation is employed to separate the desired product and permit recycling of the lower molecular weight boranes to the pyrolysis reactor.

Finally, decaborane in cyclohexane solution is readily alkylated at moderate temperatures and pressures using aluminum chloride as a catalyst:



The reaction mixture is fractionated at reduced pressures to limit reboiler temperatures and thus minimize degradation of the alkylated product. Decaborane, cyclohexane, and aluminum chloride contained in the reactor effluent are separated and recycled to the continuous alkylator.

This volume reports the fulfillment of APN's contractual obligation "to determine and recommend the most desirable sized pilot plant amenable to the process under investigation" and to submit a preliminary design.

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A diborane plant size of 12 tons per day was selected because it avoids duplication of major items of process equipment to obtain the needed throughput, and yet achieves most of the economics accruing to large scale operation. Other plant elements are sized to this output.

This total report consists of five (5) volumes, viz.:

- I. (U) Preliminary Design for a Large Scale Borane Plant
- II. (U) Theory of Diborane Pyrolysis
- III. (U) Process Development for Diborane Production
- IV. (U) Process Development for Diborane Pyrolysis
- V. (U) Process Development for Decaborane Alkylation

Volumes I, III, IV and V are security classified CONFIDENTIAL.

INTEGRATED PROCESS DESCRIPTION

The integrated process for the production of HEF-3 is represented schematically in Figure 1. It consists of the plant elements discussed in this report, enclosed within the dotted outline, together with facilities for the recovery of recycle streams and the production of the feed stocks required. Large quantities of feed materials and their handling problems inherent in their transportation and storage make it particularly advantageous to produce them in an integrated unit.

Hydrogen is prepared with a standard unit employing the steam-methane reaction at elevated temperatures and nominal pressures.

Boron trichloride is produced using proprietary process information developed by American Potash & Chemical Corporation as a commercial producer of this and other boron chemicals. Process information developed in the HEF Program by other contractors will also be evaluated and may be used. Borax and recycle borane solids are used as feed materials along with chlorine recovered by a standard commercial process from by-product hydrogen chloride. Initial and make-up amounts of chlorine will be purchased.

Ethyl chloride required in the alkylation of decaborane is produced by reacting by-product hydrogen chloride with denatured ethanol to form the alkyl chloride and water. The product is then distilled and dried. Make-up amounts of cyclohexane are required as solvent in Step II. Facilities for drying purchased material to less than 20 ppm water are included in the integrated plant.

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DESIGN CRITERIA

I. Plant Capacity and Products

The plant size of a nominal 9.5 tons per day of boron high energy fuel products was selected on the basis of achieving economies in operation without duplication of major equipment items. A larger plant would require some duplication of equipment and would not achieve significantly lower operating costs. A smaller plant would require essentially the same operating personnel and, therefore, have significantly higher operating costs.

This design provides for production of diborane which in turn is pyrolyzed to yield either pentaborane-9 or decaborane as a major product. The pyrolysis plant design permits either decaborane or pentaborane-9 to be produced in a single plant facility. In addition, it includes a step for the production of ethyl decaborane from decaborane. The production rates of these products and their purities along with the by-products produced are listed below.

Step I - Diborane Plant

B ₂ H ₆ Production Rate	11.9 tons/day
B ₂ H ₆ Purity	99% (minimum)
Yield of B ₂ H ₆ on BCl ₃	95%
HCl Production Rate	99.6 tons/day
HCl Purity	99% (minimum)

Step II - Pyrolysis Plant

a. Products During Pentaborane-9 Production

B ₅ H ₉ Production Rate	9.5 tons/day
B ₅ H ₉ Purity	99.8% (minimum)
Yield of B ₅ H ₉ on B ₂ H ₆	87.4%
B ₁₀ H ₁₄ Production Rate	0.56 ton/day
B ₁₀ H ₁₄ Purity	99% (minimum)
Yield of B ₁₀ H ₁₄ on B ₂ H ₆	5.4%
Boron Polymer Production Rate	0.68 ton/day

b. Products During Decaborane Production

B ₁₀ H ₁₄ Production Rate	9.2 tons/day
B ₁₀ H ₁₄ Purity	99% (minimum)
B ₁₀ H ₁₄ Yield on B ₂ H ₆	87.2%
Boron Polymer Production Rate	1.2 tons/day

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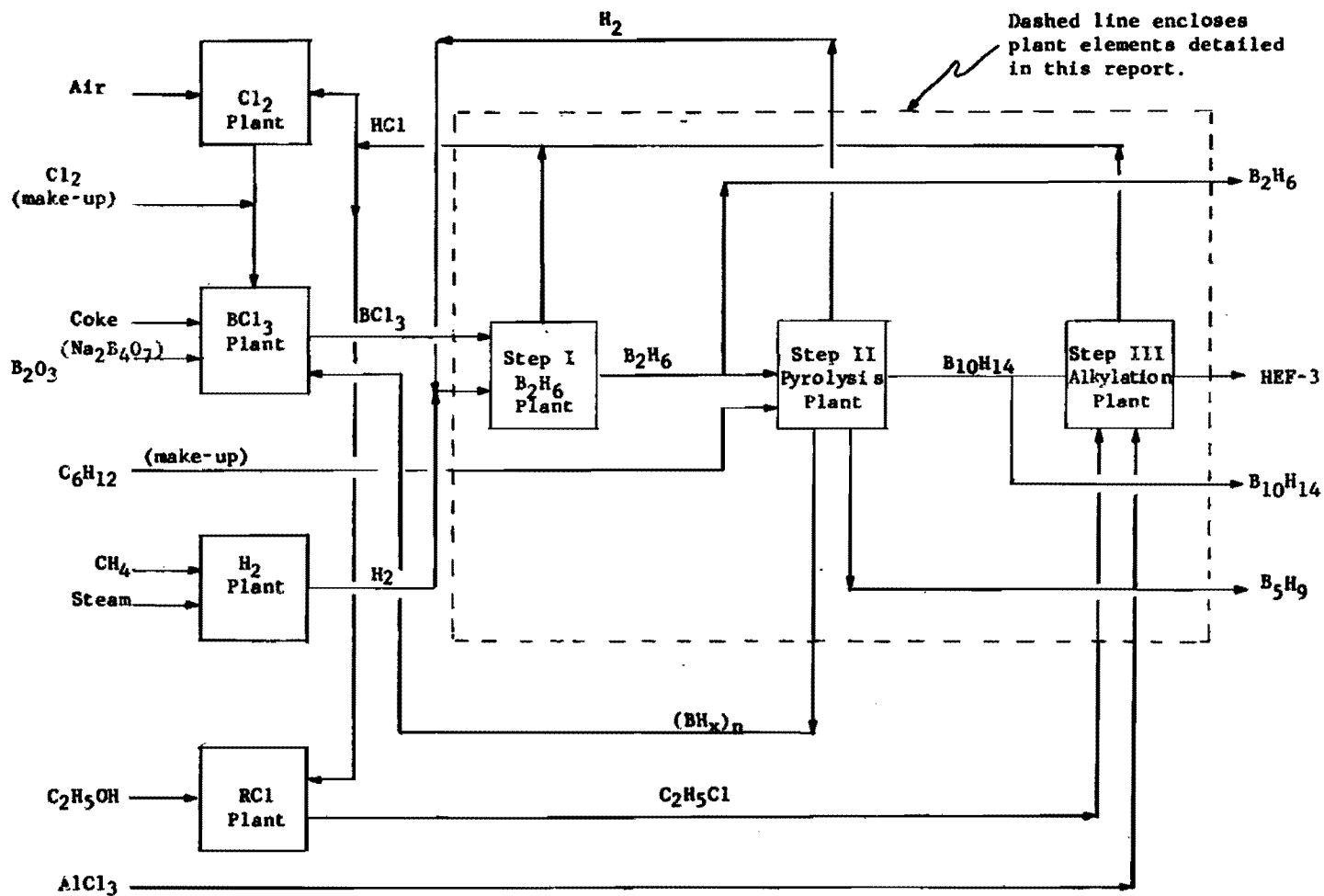


Figure 1. Integrated HEF Plant

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Step III - Alkylation Plant

HEF-3 Production Rate	9.4 tons/day
HEF-3 Yield on $B_{10}H_{14}$	98.5%
HCl Production Rate	3.2 tons/day
HCl Purity	96.0% (minimum)

HEF-3 Specification

Heating value (77°F.) defined as yielding liquid B_2O_3 and H_2O vapor.	min. 25,500 B.t.u./lb. nom. 25,800 B.t.u./lb.
Specific Gravity (77°F.)	min. 0.80 nom. 0.82
Viscosity (77°F.) (-40°F.)	max. 9 cs nom. 7 cs max. 150 cs
Vapor Pressure (77°F.)	max. 0.8 psig
Freezing Point	-76°F.
Storage Stability	Zero solids formed after 6 months storage in an inert atmosphere at temperatures from -65°F. to + 130°F.
Pyrophoricity	Non-pyrophoric from -65°F. to + 130°F.
Boiling Point at 1 atm.	min. 468°F. max. 510°F.
Chemical Analysis	min. boron 63 wt. %
Color	Water white

II. Raw Materials

Raw material specifications for the plant design presented in this report are itemized below.

1. Boron Trichloride

BCl_3	99.7% minimum purity
Phosgene	0.10% maximum
Chlorine	0.10% maximum
Silicon	300 ppm maximum

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2. Hydrogen

H ₂	99.9% minimum purity
Dew Point	< -97°F.
O ₂	5 ppm maximum

3. Cyclohexane

C ₆ H ₁₂	98% minimum purity
Commercial grade	
Dried to less than 20 ppm water	

4. Aluminum Chloride

Commercial grade through 40 mesh

5. Ethyl Chloride

USP Grade
Dried to less than 20 ppm water

III. Plant Location and Layout

The most desirable location for this plant facility is the southwestern United States, specifically southern California or southern Nevada. Factors included in this selection in descending order of importance are as follows:

1. Proximity to raw material sources.
2. Availability to railroad facilities.
3. Availability of water, electric power, fuel and means for disposal of wastes.
4. Existence of an operating organization with experience in the processing and handling of boron hydrides.
5. Manpower availability.
6. Isolation for safety, security and avoidance of nuisance.
7. Favorable topographic and climatic conditions.
8. Availability of large land areas.

The plant layout will allow for separation of administration services, warehousing facilities, raw materials receiving facilities, processing areas, process storage, final product loading facilities, maintenance

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vacilities and waste disposal. Processing units within the processing area will be further divided to permit isolation of each processing step. The degree of separation of areas will be dictated by the flammability and toxicity of raw materials, products and process streams.

IV. Safety

Because boron hydrides are toxic, pyrophoric and have wide explosive limits their processing and handling must be considered hazardous. It is notable, however, that in the AFN process none of the process streams except those in the final concentrating equipment, the storage vessels and the feed to the pyrolysis step are sufficiently concentrated to warrant unusual design treatment beyond that normally accorded hydrogen and flammable solvents. The hazards associated with the materials processed are as follows:

- Flammable - Hydrogen, ethyl chloride, dichloroborane, diborane, pentaborane, decaborane, natural gas, cyclohexane, HEF-3
- Pyrophoric - Dichloroborane, diborane, pentaborane
- Toxic - Boron trichloride, dichloroborane, diborane, pentaborane, decaborane, ethyl chloride, hydrogen chloride, HEF-3
- Corrosive - Boron trichloride, dichloroborane, hydrogen chloride

Certain provisions are therefore made for safe operation.

1. In virtually all equipment, operating pressures are positive to prevent entrance of air or water vapor and to insure that any leakage is outward. Under this condition, internal explosion due to admission of air into equipment cannot occur.
2. "Outdoor" type of construction greatly reduces the hazards of exposure of personnel to toxic gases and pockets of flammable materials. Equipment handling potentially explosive material should not be barricaded to such a degree that natural ventilation is reduced. Toxic concentrations, or large concentrations of pyrophoric or flammable materials, are thus reduced to a minimum.
3. The process system must be leaktight and must be pressure checked at normal operating temperatures.

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4. Normal process pressures range from a vacuum in the alkylation step to 300 psig pressure in the diborane step. Any excess pressure is relieved through a destructive scrubber and incinerating equipment to atmosphere. All vessels containing potentially explosive materials are protected with two parallel rupture discs and one safety relief valve. One rupture disc, backed up with a relief valve, is set to relieve at 80 per cent of the vessel design rating. This permits momentary and slight overpressures to be relieved without causing shutdown of the vessel. The parallel rupture disc is set at approximately 50 per cent higher and operates only when excessive pressure is generated as by fire or explosion.

All other vessels are furnished with a bursting disc and a back-up relief valve.

All vessels are to be constructed to meet ASME code specifications and are to be hydrostatically tested to withstand pressures of one and one-half times design ratings.

Special steel alloys are specified for vessels to be used at temperatures below -20°F. This precaution is intended to avoid embrittlement and possible fracture of the metal if the equipment is subjected to a sudden, sharp impact. The lowest temperature of any process stream is -96°F.

5. Header lines to the destructive scrubber and incinerator into which the vessel safety reliefs and blowdown vents are maintained under a constant purge of nitrogen to prevent air and moisture admittance. All of the gases vented from the diborane step pass into a destructive scrubber through a caustic solution to neutralize acid materials before being vented through an incinerator to atmosphere. Vented material from other sections of the plant not containing acid materials passes through a liquid seal directly into an incinerator before venting to atmosphere. In case of gain or loss of pressure in the header systems due to any excess relieving, the nitrogen purge is increased. In this way, any ruptured lines or vessels have nitrogen gas issuing from them into atmosphere preventing air from entering back into the equipment.
6. Diborane and pentaborane storage vessels are located away from the plant at appropriate distances from other equipment to conform to quantity-distance relationships as recommended by the U. S. Army Ordnance Manual on explosives. Barricaded enclosures would be avoided to permit natural ventilation.

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7. Enclosed operating panels for the plant are located upwind of normal prevailing winds and out of immediate process areas giving operators enough time to put on air masks or leave the area in the event of fire or sudden release of liquids or gases resulting from any equipment failure.
8. At every level of the operating structures and throughout the plant at grade level, air hoses are mounted for immediate connection to air masks.
9. Two avenues of escape are provided on all structures handling process materials: an external stairway or ladder and a fireman's type escape pole (50 ft. maximum length per section) for emergency use.
10. The plant area must be restricted and only authorized personnel permitted entry. No smoking or open fires will be permitted except in designated areas. Explosion-proof motors and push-button stations must be used throughout the plant. Switchgear in and around control rooms may be of non-explosion proof construction if located remotely or if a constant fresh air supply is forced into the room by blower to prevent flammable concentrations of gases in case of nearby leaks or spills.
11. Fire extinguishers must be located at all platform levels in structures and at grade level. In addition, several water hose stations must be located near each processing area. Storage areas will be sprinklered with activation by automatic fire sensors. It is noted, however, that no proven fire extinguishing agent exists for boron hydride fires. The best fire fighting procedure involves isolating the source of burning material from the fire and keeping the intensity of the fire down with available fire fighting equipment.

These eleven points will produce satisfactory, safe working conditions for both operators and equipment. Additional safeguards, such as fire and explosion sensors and alarms, can also be used in critical areas as additional safeguards.

V. General Design Specifications

As a means of assuring quality of materials and construction, nationally recognized codes and standards are to be utilized in the design of equipment and in the specifications for materials, equipment and services purchased. In addition, inspections are to be performed in the vendors' shops during fabrication and in the field during construction to insure conformance to those requirements.

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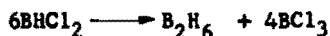
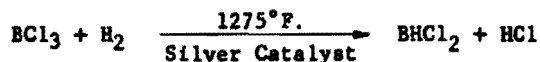
The codes and standards to be utilized include the following:

ASME Unfired Pressure Vessel Code, Section VIII, latest revision
ASA Code for pressure piping, latest revision
TEMA (Tubular Exchanger Manufacturers Association) standards
National Electric Code
NEMA (National Electrical Manufacturer's Association) standards
ASTM (American Society for Testing Materials) standards
Underwriters Laboratories standards
American Concrete Institute's code for reinforced concrete
American Institute of Steel Construction specification
Applicable state and local codes

PROCESS DESIGN

I. Diborane Production (Step I)

Diborane is prepared from boron trichloride and hydrogen according to the following reactions:



The anhydrous hydrogen chloride produced as a by-product is recycled for the production of boron trichloride.

The process may be divided in principal phases as follows:

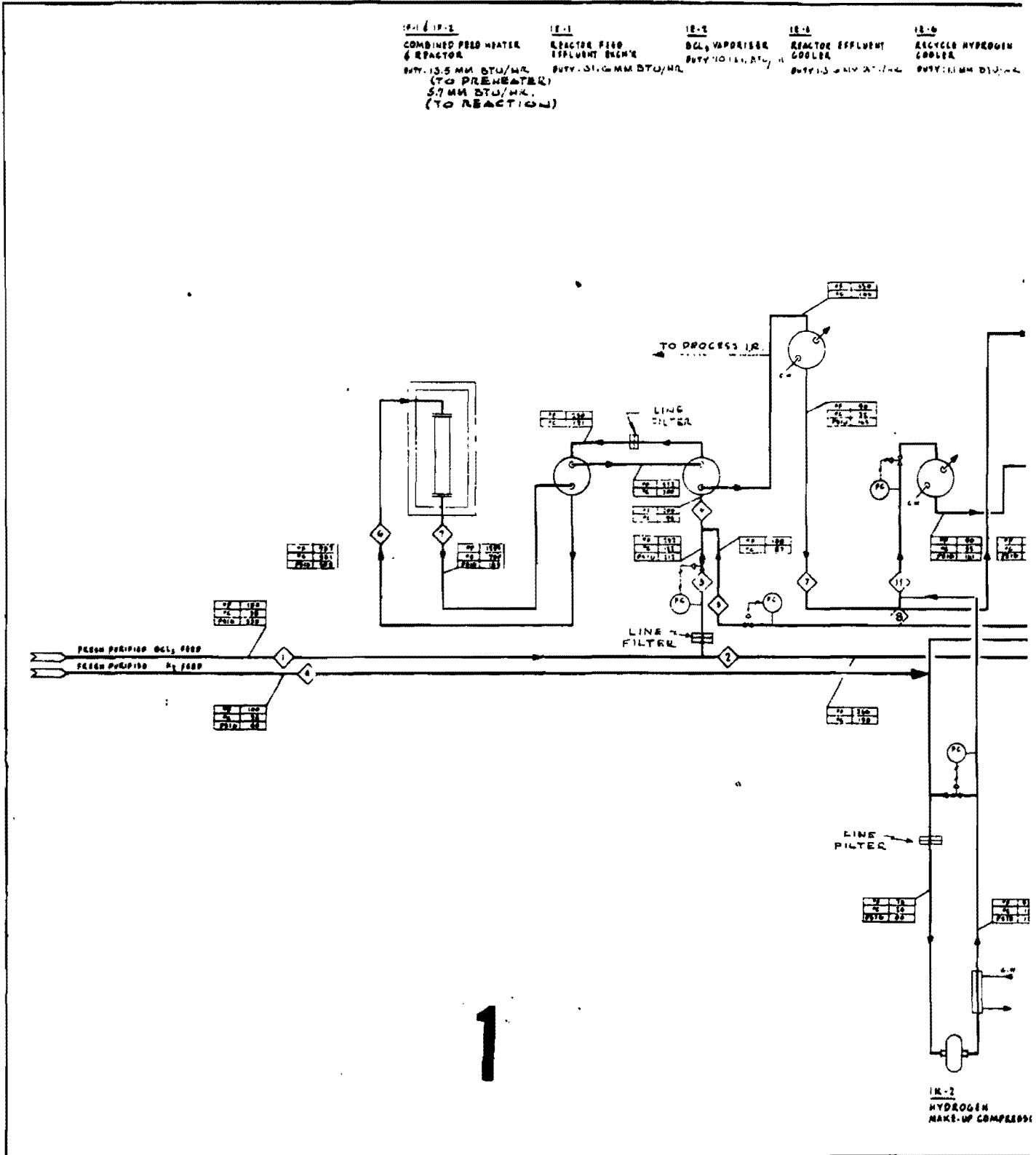
- A. Hydrogenation of Boron Trichloride
- B. Separation of Reactor Products
- C. Disproportionation
- D. Hydrogen Chloride Recovery

These process steps are shown in the flow diagram presented in Figure 2 and material balance data are given in Table 1.

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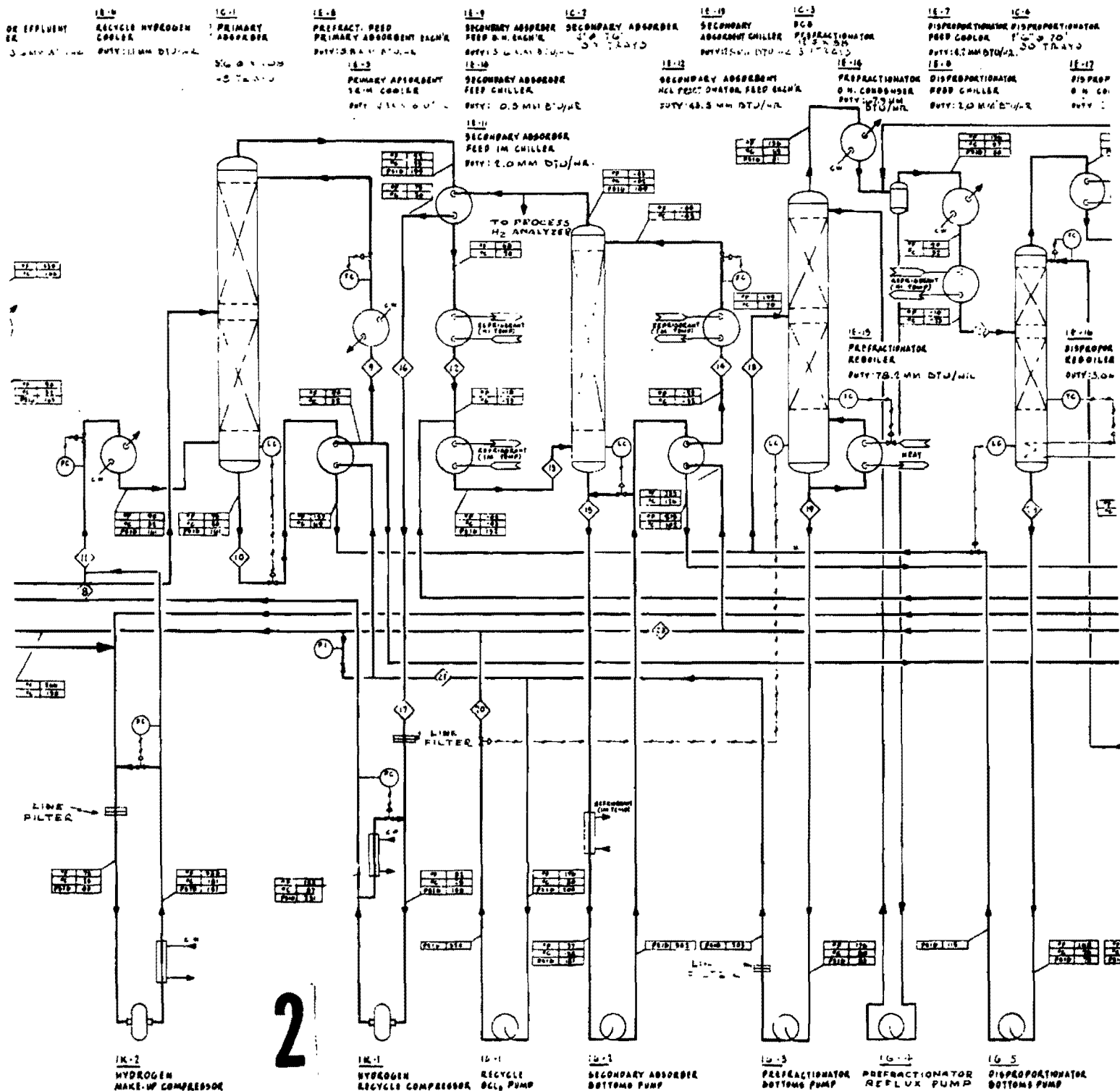
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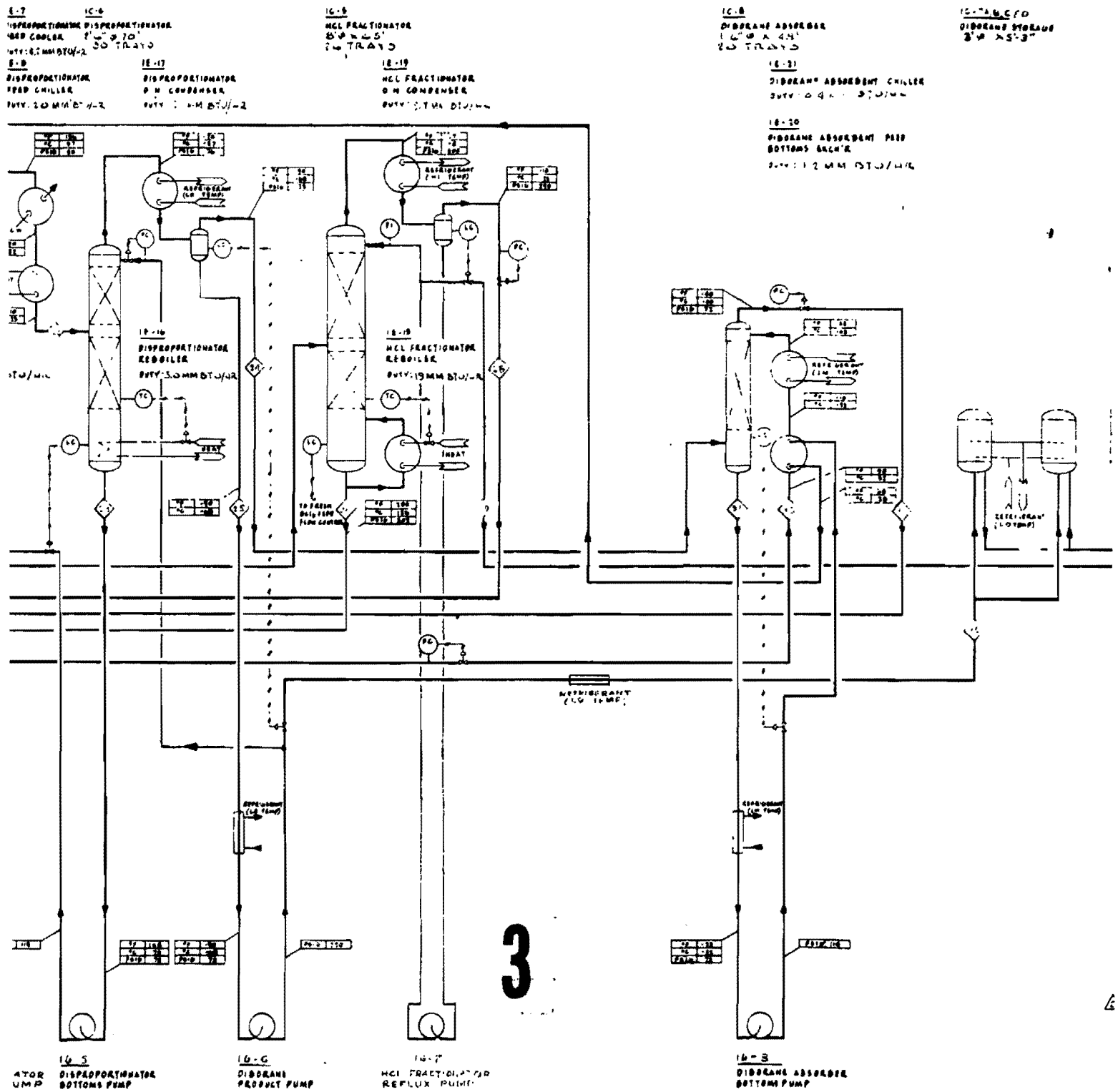
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TABLE 1
Diborane Production (Step I) Material Balance
 (moles/hour)

Stream	Stream No.	H ₂	B ₂ H ₆	HCl	BHCl ₂	BCl ₃	Total
Fresh BCl ₃ Feed	1					75	75
Recycle BCl ₃ Feed	2					1,065	1,065
Total BCl ₃ to Reactor	3					1,140	1,140
Fresh H ₂ Feed	4	228					228
Total H ₂ to Reactor	5	3,420					3,420
Reactor Charge	6	3,420				1,140	4,560
Reactor Products	7	3,190		228	228	912	4,558
Recycle Stripping H ₂	8	6,440		8,320	18,850	106,900	140,510
Absorbent to Primary Absorber	9					3,490	3,490
Primary Absorber Bottoms	10	42			228	3,530	3,800
Total Stripping H ₂	11	1,400					1,400
Primary Absorber Overhead	12	4,550		228		883	5,661
Total Feed to Secondary Abs.	13	4,575		277		884	5,736
Absorbent to Secondary Abs.	14					4,950	4,950
Secondary Abs. Bottoms	15	28		277		5,780	6,085
Secondary Abs. Overhead ^(a)	16	4,550					4,550
Total Gas to H ₂ Compressor	17	4,550					4,550
Total Prefract. Feed	18	42			253	3,930	4,225
Total Prefract. Bottoms	19					4,100	4,100
Prefract. Bottoms to Reactor	20					181	181

(a) May contain 12.6 mph BCl₃
 stripped from absorber

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TABLE 1 (Cont.)

Diborane Production (Step I) Material Balance
(moles/hour)

Stream	Stream No	H ₂	B ₂ H ₆	HCl	BHCl ₂	BCl ₃	Total
Prefract. Bottoms to 1C-1 & 1C-8	21					3,918	3,918
Disproportionator Feed	22	41.9	54.5		253	253	602.4
Disproportionator Bottoms	23				25.2	406	431.2
B ₂ H ₆ Absorber Feed	24	41.9	54.5				96.4
B ₂ H ₆ Liquid Product	25		35.84				35.84
Total HCl Fract. Bottoms	26					5,780	5,780
HCl Fract. Bottoms to Reactor	27					884	884
HCl Fract. Ohd. Vapor Product	28	28.0		49.0			77
HCl Liquid Product	29			228.0			228
Absorbent to B ₂ H ₆ Abs.	30					4,200	4,200
B ₂ H ₆ Absorber Bottoms	31		54.6			420	474.6
B ₂ H ₆ Absorber Ohd. Vapor	32	42					42

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A. Hydrogenation of Boron Trichloride

The design of the reactor section is based on several factors derived from laboratory and prepilot plant data and confirmed by pilot plant experience:

1. The reaction at 1275°F. is rapid in the presence of silver as a catalyst. The reactor is designed with a residence time of 0.5 second.
2. Silver, in addition to acting as a catalyst, is utilized as a material of construction for equipment items handling hot hydrogen-boron trichloride mixtures, which are extremely corrosive toward the more usual materials of construction. In addition, hydrogen and boron trichloride are premixed before heating above 500°F. inasmuch as hot boron trichloride by itself can be corrosive to silver.
3. At a hydrogen to boron trichloride ratio of 3:1, 20 per cent of the boron trichloride fed to the reactor is converted to dichloroborane at a temperature of 1275°F. Higher boron trichloride conversions can be obtained at higher hydrogen to boron trichloride ratios, e.g., 39 per cent at 14:1, but such conditions are economically less favorable because of the inordinately greater volumes of gas to be handled. Slightly higher conversions can also be obtained at higher temperatures, but at an increasing danger of converting some of the boron trichloride to elemental boron and of damaging the silver (melting point 1760°F.).
4. The reactor effluent is rapidly cooled to "freeze" the favorable equilibrium existing at high temperatures. Slow cooling would permit back reaction, seriously reducing the conversion.

In practice, make-up boron trichloride is added to that recycling from the separation section of the plant, and after passing through a polishing filter (1V-1) is mixed with recycle hydrogen from the top of the secondary absorber (1C-2). A compressor (1K-1) provides the energy to move the hydrogen and boron trichloride through the reactor section which operates at approximately 200 psig. The reaction mixture proceeds through exchangers (1E-1 and 1E-2) where heat exchange with the reactor effluent vaporizes the boron trichloride and heats the reactant gases. All parts of exchanger 1E-1 exposed to process gases are lined (not plated) with 1/16 inch silver. The gas mixture is filtered prior to entering this exchanger to insure a clean feed to the reactor. A combination preheater-reactor (1F-1, 1F-2), gas-fired, brings the reaction mixture up to a

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temperature of 1275°F. and holds it there for the required 0.5 second. While both the preheater and reactor are silver lined, only the reactor contains silver catalyst screens.

The silver lined exchanger (1E-1) provides rapid initial cooling of the reactor effluent, thus "freezing" the high temperature equilibrium, and further cooling is accomplished in exchangers 1E-2 and 1E-4. The latter uses plant cooling water. Performance of the reactor is continuously monitored by an infrared analyzer which determines the dichloroborane content of the reactor effluent. This mixture, containing H_2 , BCl_3 , $BHCl_2$ and HCl in the mole ratio of 2.8:0.8:0.2:0.2, proceeds to the separation system.

B. Separation of Reactor Products

A physical absorption process which takes advantage of the substantial differences in volatility among hydrogen, hydrogen chloride and dichloroborane is utilized to effect the separation of the reactor products. An important feature which greatly simplifies the system is that boron trichloride, already present as a reactant, is used as the absorbing medium.

The cooled reactor effluent, a two-phase stream, is fed to the center of the primary absorber (1C-1). Liquid boron trichloride at 90°F. and 160 psig enters the top of the column to act as the absorbent for $BHCl_2$ while hydrogen is introduced at the bottom to strip out any hydrogen chloride dissolved in liquid coming down the column. Thus the upper section of the primary absorber (1C-1) is a dichloroborane absorber, and the lower section is a hydrogen chloride stripper. Since upon disproportionation six molecules of dichloroborane form only one molecule of diborane, the effect of contamination of the column bottoms with HCl , which eventually comes out with the diborane product, is multiplied. It is therefore important that the stripping gas, composed of both recycle and make-up hydrogen, be free of hydrogen chloride.

The overhead from the primary absorber, containing hydrogen, hydrogen chloride and boron trichloride vapor, is chilled and fed to the bottom of the secondary absorber (1C-2) which operates at -45°F. and 150 psig. Again liquid boron trichloride is the absorbent. Whereas hydrogen chloride passed overhead in the primary absorber (1C-1), it is now absorbed in the secondary absorber (1C-2) at the lower temperature, leaving only hydrogen with a small amount of boron trichloride to go overhead. This hydrogen is monitored with a purity analyzer which indicates the effectiveness of the secondary absorber (1C-2) in removing hydrogen chloride and therefore the suitability of this stream for stripping gas in the primary absorber (1C-1). The bulk of this hydrogen is recycled back to the catalytic reactor by way of compressor (1K-1).

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C. Disproportionation

The bottoms of the primary absorber (1C-1), containing dichloroborane and boron trichloride, are fed to the dichloroborane prefractionator (1C-3). This distillation column has two functions: (1) it concentrates dichloroborane at the top of the column where disproportionation is promoted through the mass action principle, and (2) it provides a pure boron trichloride bottoms product which is used as absorbent and as recycle feed to the hydrogenation reactor (1F-1, 1F-2).

The column feed also contains some dissolved hydrogen. As this non-condensable gas passes through the water-cooled, overhead condenser it carries with it the diborane formed by disproportionation and equilibrium vapor pressure concentrations of dichloroborane and boron trichloride. This mixture, after being chilled, constitutes the feed to the disproportionator (1C-4).

Disproportionation is completed in this column, and diborane is fractionated from dichloroborane (and boron trichloride) to produce a pure liquid product which is pumped (1C-6) to refrigerated storage tanks (1C-7A and 1C-7B). The column bottoms may contain some dichloroborane and are therefore returned to the prefractionator (1C-3).

The rapid polymerization of diborane at elevated temperatures is well known; in addition, pilot plant experience has indicated that dichloroborane may similarly polymerize to form solids containing boron, hydrogen and chlorine. For this reason the disproportionator section is operated at lowest pressure in the diborane plant, nominally 75 psig. This places the bottoms temperature of the prefractionator (1C-3), the highest in this section, at 176°F. Under this circumstance very little solids are formed and these are well controlled by a small line filter in the discharge of the prefractionator bottoms pump (1C-3). This pressure also establishes the refrigeration level required for the condensation of diborane at the top of the disproportionator (1C-4) at -100°F. This level of refrigeration is practical, but decidedly lower temperatures would be considerably more difficult and costly to achieve.

Hydrogen, dissolved in the prefractionator (1C-3) feed, exits from the disproportionator (1C-4) accumulator as a non-condensable gas and carries with it diborane vapor. This stream is fed to the diborane absorber (1C-8) where the diborane is picked up in cold (-45°F.) boron trichloride giving an overhead stream of hydrogen for recycle. This gas, after compression (1K-2), makes up a portion of the stripping gas for the primary absorber (1C-1). The absorber (1C-8) bottoms are recycled to the prefractionator (1C-3) reflux stream.

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D. Hydrogen Chloride Recovery

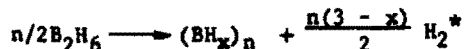
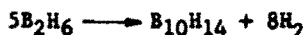
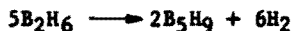
The mixture of hydrogen chloride and boron trichloride constituting the secondary absorber (1C-2) bottoms is fed to the hydrogen chloride fractionator (1C-5). Since these compounds are both stable at elevated temperatures, the distillation column can be operated at 300 psig, giving a bottoms temperature of 285°F. At this pressure hydrogen chloride condenses at 17°F., and high temperature refrigerant (-20°F.) can be used in the condenser. The anhydrous liquid by-product hydrogen chloride is taken off to refrigerated storage tanks and is eventually recycled to make boron trichloride in an auxiliary plant. The pure boron trichloride bottoms from the hydrogen chloride fractionator (1C-5) are used both for feed to the hydrogenation reactor (1F-1, 1F-2) and, after chilling, for absorbent in the secondary absorber (1C-2).

Non-condensable gas (H₂) vented from the accumulator contains hydrogen chloride vapor and is therefore fed to the bottom of the secondary absorber (1C-2).

II. Pentaborane/Decaborane Production (Steps II and IIA)

A. Diborane Pyrolysis (Step II)

The pyrolysis of diborane involves a series of complex reactions, reported in Volume Two, which, in terms of principal products, can be reduced to the following net reactions:



Other boron hydrides, including tetraborane, pentaborane-11 and a number of very active species are also formed during diborane pyrolysis. Pentaborane-9, when allowed to continue reacting with diborane, forms decaborane which, in turn, leads to the formation of boron hydride polymer upon further reaction with diborane.

This plant is designed to obtain maximum conversion of diborane to either pentaborane-9 or decaborane, depending on the product in greatest demand, under slightly different conditions of operation. As a necessary corollary, the production of borane polymer, which can be utilized only by recycling to an early step of the integrated process, is held to a minimum.

* For material balance purposes in this report,
n = 10 and x = 1.

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Material balances for both the pentaborane-9 and the decaborane operations are presented in Tables 2 and 3, respectively. Figure 3 shows the process flows.

Maximum yield of the desired product is obtained through the selection of appropriate operating conditions for the reactor (2R-1). Since this is the only equipment item in which such conditions exist, it is essential that pyrolysis be avoided in other units. This objective is accomplished by (1) rapid quenching of the reactor effluent to 100°F., at which temperature the pyrolysis rate is negligible, and (2) separating the unreacted diborane and lower intermediates, such as tetraborane and pentaborane-11, from the reaction products at low temperatures in the final purification steps. Cyclohexane, which is virtually inert in the pyrolysis system, is used as a carrier solvent and quenching medium. Since it is also the solvent used in alkylation of decaborane (Step III), the pyrolysis plant produces a 20 mole per cent cyclohexane solution of decaborane. If a solid product is desired, this solution is processed in the decaborane crystallization unit (Step IIA).

Step II is comprised of three elements:

1. Pyrolysis
2. Separation of Pyrolysis Products
3. Diborane Recovery from Hydrogen Off-Gas

These are discussed in detail for the pentaborane-9 process, and then the significant differences in the decaborane process are pointed out.

1. Pyrolysis

The reactor conditions established for the production of pentaborane-9 are the following:

Temperature	-	500°F.
Pressure	-	75 psig
Reactor Retention Time*	-	2 seconds
B ₂ H ₆ in Feed Gas	-	12 mole per cent

A preheater (2E-1) raises the temperature of the recycle gases going to the reactor (2R-1) to 200°F. Following the addition of fresh diborane feed from Step I, the gas mixture enters a jacketed reactor where the heating cycle is rapidly completed. Heat is furnished to the jacket by means of a hot Aroclor (TM, Monsanto Chemical Co.) circulating system. During the two second reactor retention time a small increment of pyrolysis takes place after which the reactor effluent is quenched by a 90°F. stream, predominantly cyclohexane, from the phase separator (2PH-1).

* At reactor conditions.

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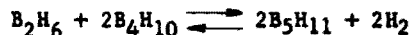
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The combined liquid-gas stream passes through a heat exchanger (2E-2) to cool it back down to 90°F. and then discharges into the phase separator (2PH-1). Reactor recycle compressor (2K-1) sends the gas stream from the phase separator back through the reactor, while the liquid is recycled as quench liquor by a pump (2G-1). The pyrolysis products accumulating in the liquid phase are continuously drawn off in a small stream fed to the separation section.

Since hydrogen is generated in the pyrolysis reactions, the pressure of the reactor system tends to rise. However, a pressure controller relieves gas from this section to the diborane absorber (2C-1) where the borane values in the gas are recovered.

2. Separation of Pyrolysis Products

Liquid withdrawn from the recirculating reactor liquid stream is centrifuged (2V-1) to remove the small amount of insoluble borane polymer formed in the reactor and then fed to the B₂ stripper No. 1 (2C-3). Here stripping hydrogen from the B₂ absorber (2C-1) strips diborane and a small amount of tetraborane from the stream at 5 psig and 100°F. The off-gas from the stripper is added to the process recycle gas stream, which is compressed (2K-2) and returned to the reactor system. The bottoms liquid is fed to B₂ stripper No. 2 (2C-4) operating at 120°F. and 5 psig. In this column most of the remaining tetraborane and also pentaborane-11 are removed from the liquid. This action is facilitated by the reconversion of pentaborane-11 to tetraborane and diborane according to the equilibrium reaction:



Removal of diborane and tetraborane, both more readily stripped than B₅H₁₁, as well as the presence of excess hydrogen from the stripping gas, keeps this reaction proceeding to the left. The overhead gas from this column is also added to the process recycle gas stream.

The bottoms liquid from B₂ stripper No. 2 (2C-4) is fed to the B₅ distillation column (2C-5) which provides a bottoms stream of decaborane dissolved in cyclohexane containing no pentaborane-9. This stream, after being centrifuged (2V-2) to remove any additional solids which may have formed, is fed to the C₆ distillation column (2C-6) to make the desired final decaborane solution of 20 mole per cent. The overhead from this column, pure cyclohexane, is used as wash solvent for the various centrifuge cakes and as part of the absorbent in the diborane absorber (2C-1).

The overhead of the B₅ column (2C-5) is 30 mole per cent B₅H₉ in cyclohexane and is fed to the B₅ concentrating column (2C-7). Here the remaining cyclohexane is separated as a bottoms product which is recycled to the B₅ column (2C-5) for recovery of its B₅H₉ content. The

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TABLE 2
PENTABORANE PRODUCTION (STEP II)

Material Balance
(mole/hr)

	STREAM NO	H ₂	B ₂ H ₆	B ₅ H ₉	B ₄ H ₁₀	B ₅ H ₁₁	B ₁₀ H ₁₄	(BH) _x ₁₀	C ₆ H ₁₂	C ₅ H ₁₂	TOTAL
Reactor Feed	1		35.85								35.85
Liquid Recycle to Reactor	2	0.65	5.43	0.43	0.07	0.18	0.01		326.43		333.20
Reactor Recycle Gas	3	4754	612	20	4	8	8		210		5616.00
Reactor Liquid	4	1.20	5.17	14.62	0.61	0.88	0.37	0.37	338.69		361.91
Reactor Gas to Absorber	5	104.99	13.42	0.45	0.07	0.19			4.66		123.78
Liquid to Stripper No. 1	6	1.20	5.17	14.62	0.61	0.88	0.37		338.69		361.54
Gas to Stripper No. 1	7	18.18	0.07						0.25		18.50
Liquid from Stripper No. 1	8	0.22	0.18	14.24	0.54	0.86	0.37		333.88		350.29
Gas from Stripper No. 1	9	19.16	5.06	0.38	0.07	0.02			5.06		29.75
Gas to Stripper No. 2	10	18.18	0.07						0.25		18.50
Liquid from Stripper No. 2	11	0.22	0.06	13.57	0.11	0.16	0.37		326.18		340.67
Gas Off Stripper No. 2	12	18.18	0.19	0.67	0.43	0.70			7.95		28.12
Gas Off B ₅ Dist. Column	13	0.57	0.04	0.31	0.01				0.31		1.24
Overhead Liquid B ₅ Dist. Col.	14	0.02	0.02	13.28	0.05	0.07			28.82		42.26
Bottoms B ₅ Dist. Col.	15			0.05	0.39	0.02		0.10	297.06		297.62
Bottoms from C ₆ Column	16						0.38	0.01	1.56		1.95
Overhead Gas C ₆ Col.	17	0.02							0.02		0.04
Overhead Liquid C ₆ Col.	18	0.12					0.01		295.48		295.61
Overhead Gas B ₅ Conc. Col.	19	0.44	0.02	0.60							1.86
Bottoms B ₅ Conc. Col.	20			0.04			0.01	0.07	28.81		28.93
Overhead Liq. B ₅ Conc. Col.	21	0.01	0.01	12.55					0.01	0.25	12.83
Bottoms B ₅ Prod. Col.	22			12.53					0.01	0.01	12.55
Overhead Gas to Flare	23	0.01	0.01	0.02						0.24	0.28
9, 12, 13, 17, 19, 32 Recycle	24	54.57	18.46	1.98	0.09	0.04			17.20		92.34
Gas Off 1st K.O. Pot	25	54.56	18.34	0.77	0.07	0.02			3.86		77.62
Liquid Off 1st K.O. Pot	26	0.01	0.12	1.21	0.02	0.02			13.34		14.72
Gas Off 2nd K.O. Pot	27	54.55	18.21	0.16	0.04				0.39		73.35
Liquid from 2nd K.O. Pot	28	0.01	0.13	0.61	0.03	0.02			3.47		4.27
Absorber Off Gas	29	52.91	0.16						0.84		53.91
Absorber Liquid Out	30	1.94	16.27	1.29	0.20	0.51	0.02		978.05		998.28
Absorber Liquid from Stripper	31	0.65	0.19	0.83	0.12	0.34			648.54		650.67
Stripper Off Gas	32	15.19	10.70	0.03	0.01	0.02			3.78		29.73
Stripping Gas to Abs. Stripper	33	14.54	0.06						0.21		14.81
C ₆ H ₁₂ Fresh Feed	34								2.90		2.90
Solid Product Out	35							0.48			0.48

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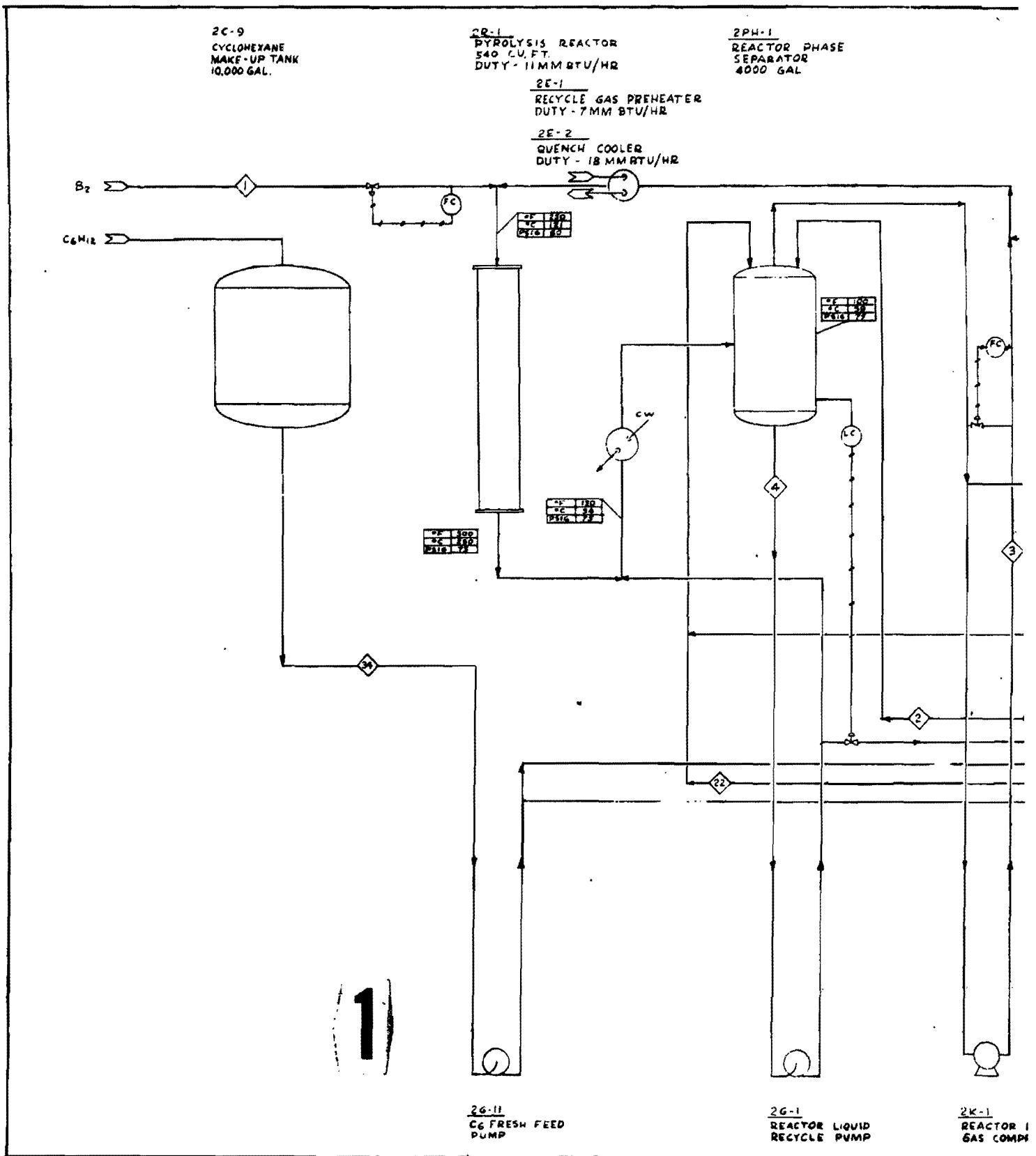
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TABLE 3
DECABORANE PRODUCTION (STEP II)

Material Balance
(mole/hr)

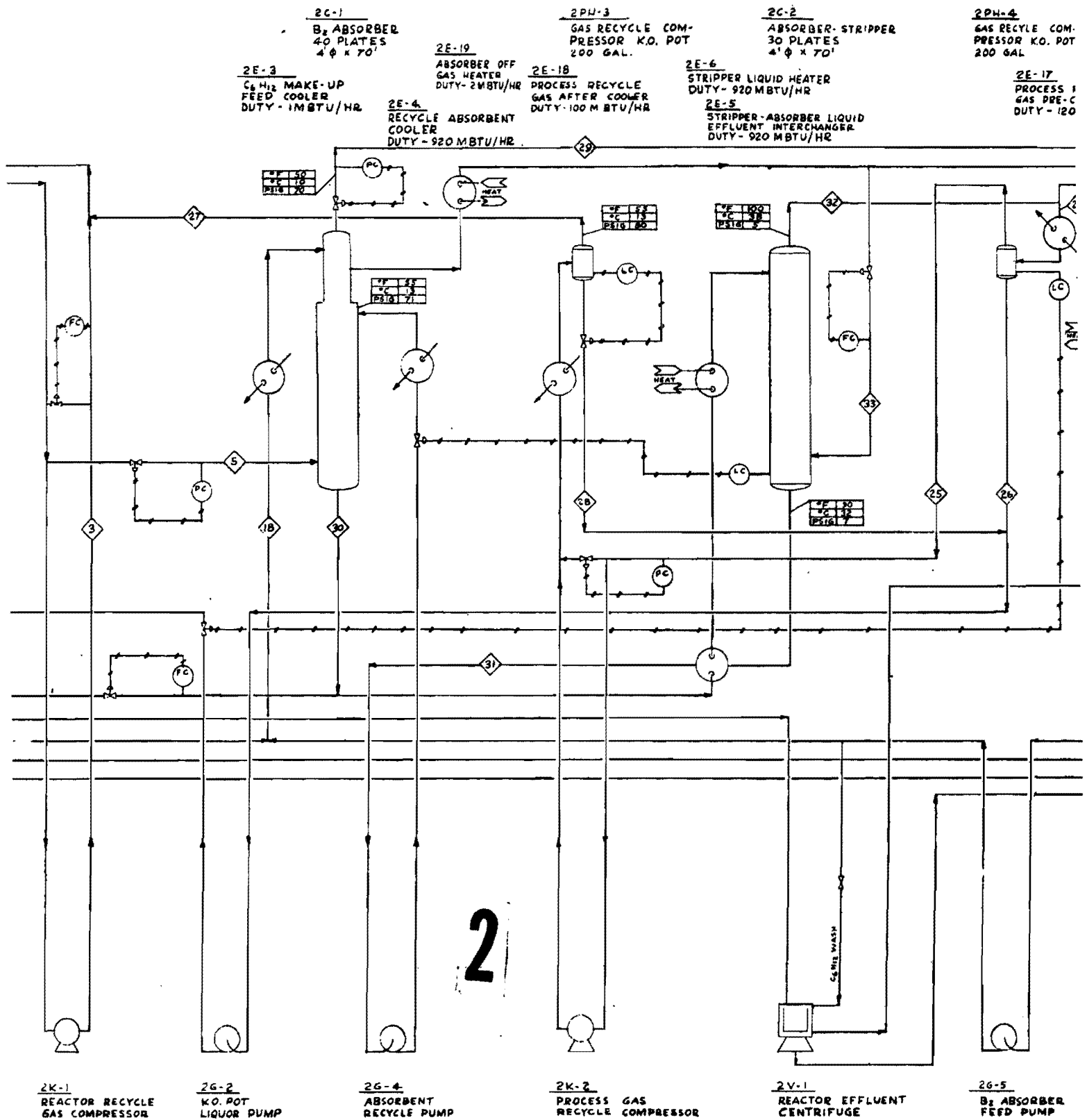
	STREAM NO.	H ₂	B ₂ H ₆	B ₅ H ₉	B ₄ H ₁₀	B ₅ H ₁₁	B ₁₀ H ₁₄	(BH _x) ₁₀	C ₅ H ₁₂	C ₅ H ₁₁	TOTAL
Reactor Feed	1		35.85								35.85
Liquid Recycle to Reactor	2	0.06	0.41	3.18	0.03	0.15			23.00		26.83
Reactor Recycle Gas	3	4750	608	215	3	8			215		5584.00
Reactor Liquid	4	0.145	0.321	14.62	0.045	0.622	6.26	0.49	26.25		48.312
Reactor Gas to Absorber	5	110.23	14.15	5.00	0.07	0.19			5.00		134.64
Liquid to Stripper No. 1	6	0.145	0.321	14.62	0.045	0.622	6.26		26.25		48.263
Gas to Stripper No. 1	7	18.18	0.07						0.25		18.50
Liquid from Stripper No. 1	8	0.04	0.015	14.26	0.04	0.61	6.26		25.30		46.525
Gas from Stripper No. 1	9	18.285	0.376	0.36	0.005	0.012			1.20		20.238
Gas to Stripper No. 2	10	18.18	0.07						0.25		18.50
Liquid from Stripper No. 2	11A	0.142	0.024	13.57		0.116	6.26		23.69		43.802
Gas Off Stripper No. 2	12	17.066	1.32	0.69	0.008	0.004			1.86		20.948
B ₅ Conc. Col. Ovhd. Gas	19	3.20	0.15	0.286					0.007		3.508
B ₅ Conc. Col. Bottoms	20A			0.002			6.26	0.37	23.00		29.632
B ₅ Conc. Col. Ovhd. Liq.	21	0.13	0.01	12.658					0.683	0.25	13.731
Bottoms B ₅ Prod. Col.	22A			12.64					0.683	0.01	13.333
Ovhd. Gas to Flare	23	0.13	0.01	0.02					0.01	0.24	0.41
9, 12, 19, 32 Gas Recycle	24	53.74	15.69	3.02	0.05	0.06			6.32		78.88
Gas Off 1st K.O. Pot	25	53.73	15.52	0.92	0.04	0.03			3.90		74.14
Liquid Off 1st K.O. Pot	26	0.01	0.17	2.10	0.01	0.03			2.42		4.74
Gas Off 2nd K.O. Pot	27	53.72	15.39	0.22	0.02	0.01			0.39		69.75
Liquid Off 2nd K.O. Pot	28	0.01	0.13	0.70	0.02	0.02			3.51		4.39
Absorber Off Gas	29	58.55	0.17						0.86		59.58
Absorber Liquid Out	30	2.10	13.98	108.00	1.01	5.10			782.00		912.19
Absorber Liquid from Stripper	31	1.39	0.14	106.18	0.97	5.06				758.79	872.53
Stripper Off Gas	32	15.19	13.84	1.82	0.04	0.04			3.25		34.18
Stripping Gas to Abs. Stripper	33	14.54	0.06						0.21		14.81
C ₅ H ₁₂ Fresh Feed	34								26.10		26.10
Solid Product	35							0.86			0.86
Deca to Crystallization	36						6.26		25.04		31.30

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2PH-4

GAS RECYCLE COM-
PRESSOR K.O. POT.
200 GAL

2E-17

PROCESS RECYCLE
GAS PRE-COOLER
DUTY - 120 MBTU/HR

2C-3

B₂ STRIPPER NO.1
50 PLATES
4' Ø x 110'

2E-7

B₂ STRIPPER NO.1
LIQUID HEAT EXCHANGER
DUTY - 450 MBTU/HR

2C-4

B₂ STRIPPER NO.2
50 PLATES
4' Ø x 110'

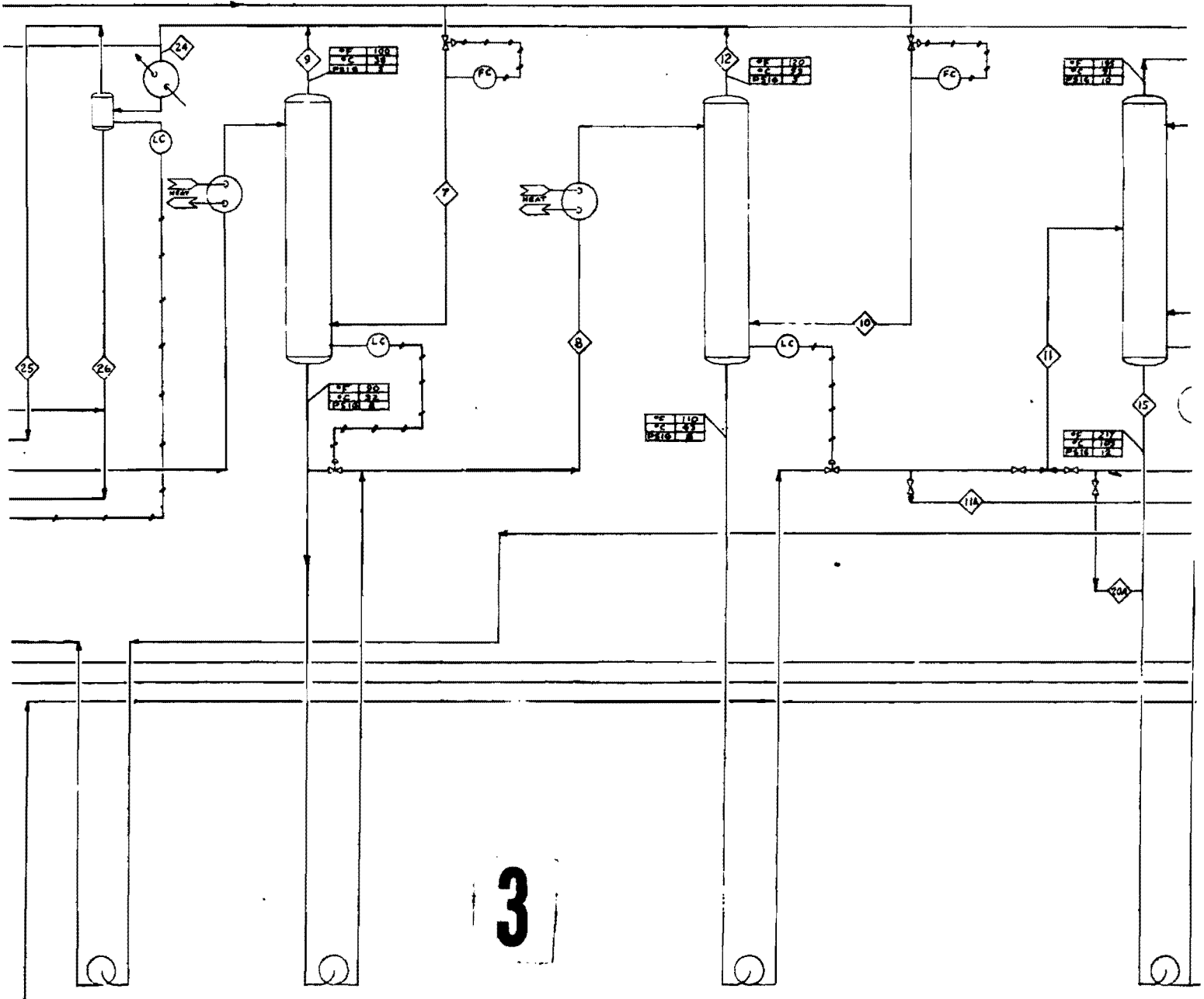
2E-8

B₂ STRIPPER NO.2
LIQUID HEAT EXCHANGER
DUTY - 600 MBTU/HR

2C-5

B₂ DISTILL
20 PLATE
3' Ø x 30'

2
B
D



3

26-5

B₂ ABSORBER
FEED PUMP

26-6

B₂ STRIPPER NO.2
FEED PUMP

26-7

B₂ STRIPPER NO.2
EFFLUENT PUMP

26-8

C₆ COLUMN
FEED PUMP

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2C-5
B₅ DISTILLATION COLUMN
20 PLATES
3' Ø x 30'

2E-10
B₅ DISTILLATION COLUMN CONDENSER
DUTY - 30 MM BTU/HR

2E-9
B₅ DISTILLATION COLUMN REBOILER
DUTY - 42 MM BTU/HR

2C-6
C₆ DISTILLATION COLUMN
9 PLATES
8' Ø x 34'

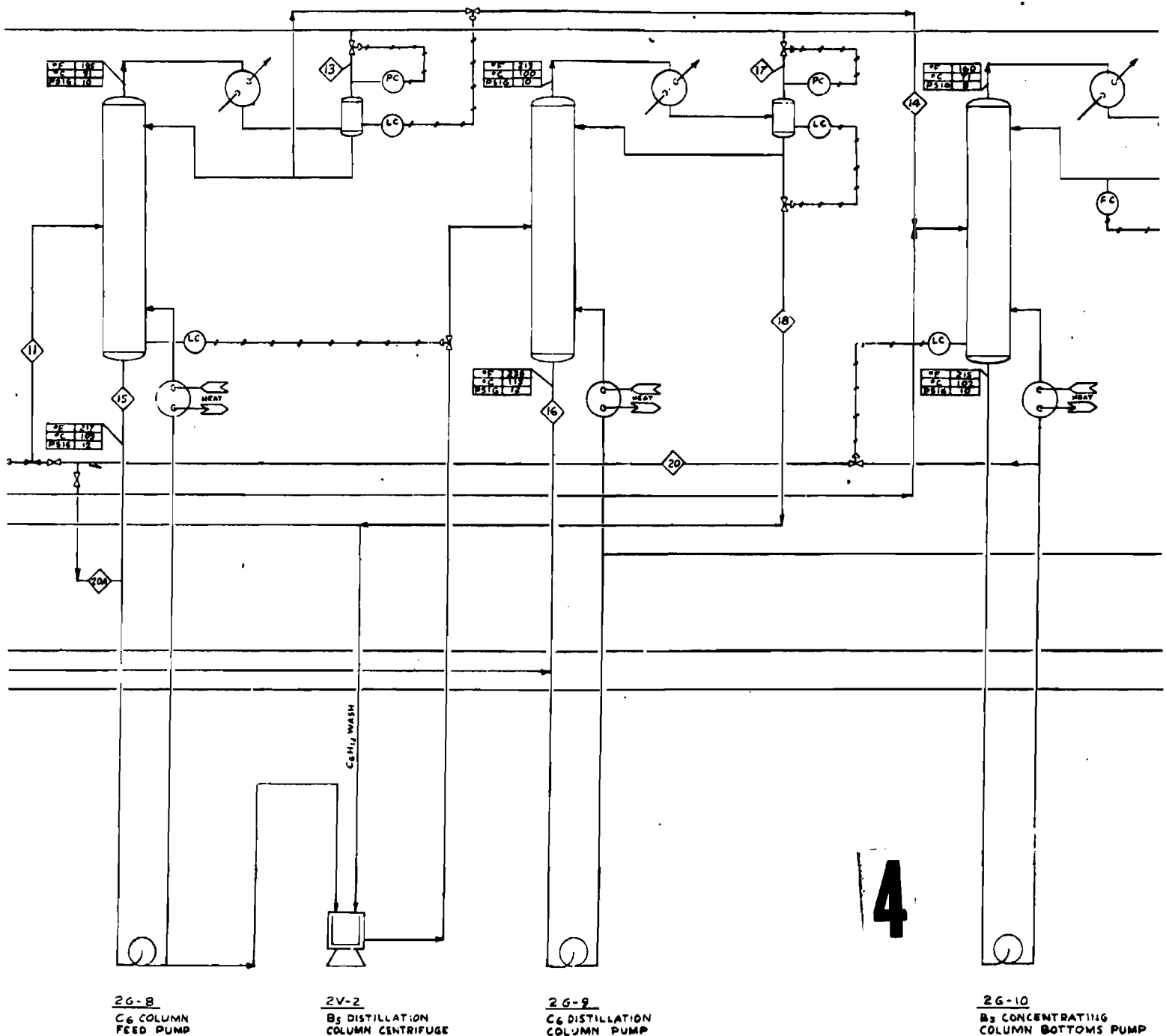
2E-12
C₆ DISTILLATION COLUMN CONDENSER
DUTY - 13 MM BTU/HR

2E-11
C₆ DISTILLATION COLUMN REBOILER
DUTY - 15 MM BTU/HR

2C-7
B₅ CONCENTRATING COLUMN
40 PLATES
2' Ø x 90'

2E-14
B₅ CONCENTRAT
DUTY - 700 M

2E-13
B₅ CONCENTRATING CC
DUTY - 780 M BTU/H



2G-8
C₆ COLUMN
FEED PUMP

2V-2
B₅ DISTILLATION
COLUMN CENTRIFUGE

2G-9
C₆ DISTILLATION
COLUMN PUMP

2G-10
B₅ CONCENTRATING
COLUMN BOTTOMS PUMP

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2C-7
B₂ CONCENTRATING COLUMN
40 PLATES
2' Ø x 90'

2C-8
B₂ PRODUCT COLUMN
30 PLATES
1' Ø x 70'

2C-10
PENTABORANE STORAGE
5000 GAL

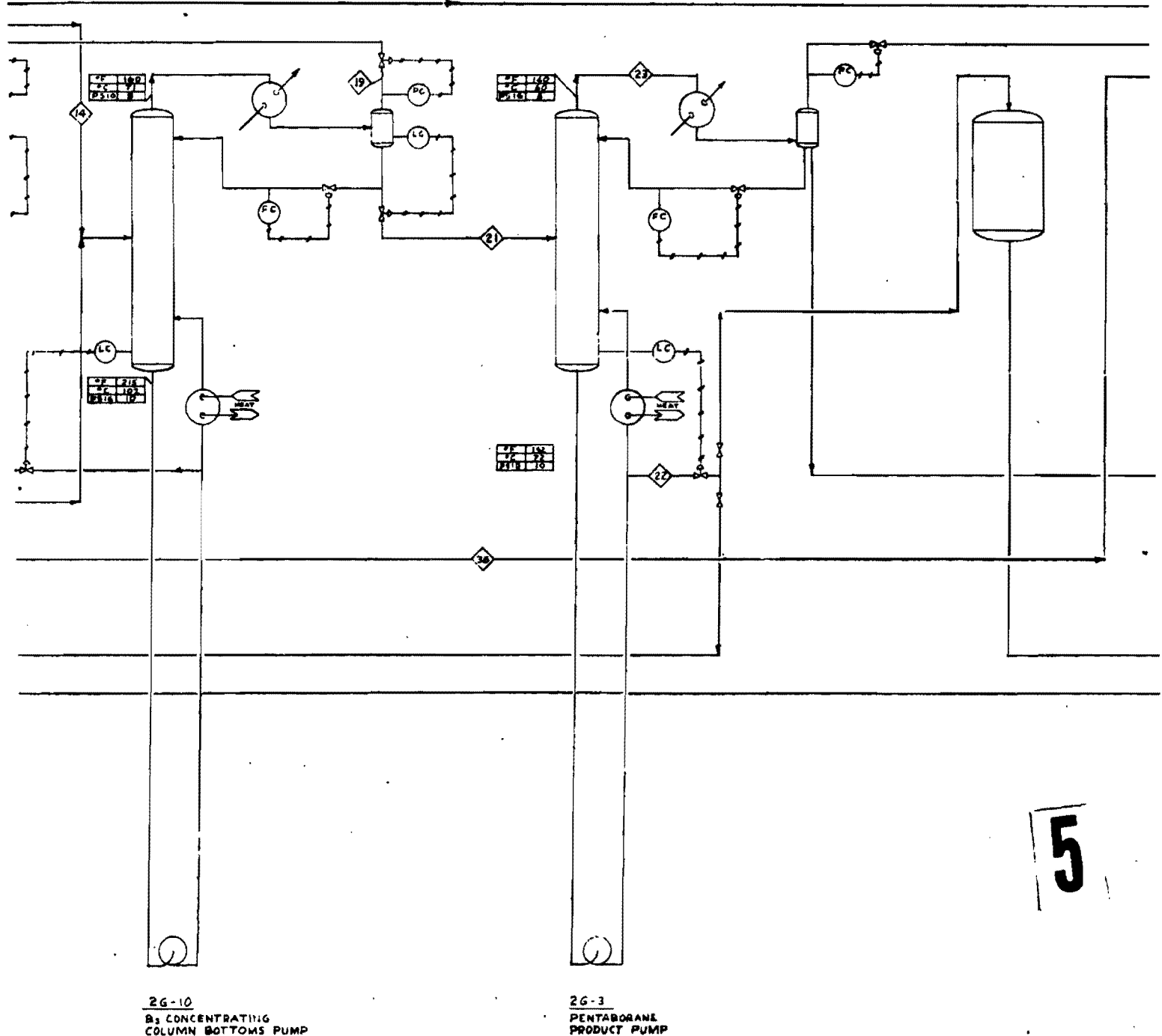
2E-14
B₂ CONCENTRATING COLUMN CONDENSER
DUTY - 700 M BTU/HR

2E-16
B₂ PRODUCT COLUMN CONDENSER
DUTY - 100 M BTU/HR

2E-13
B₂ CONCENTRATING COLUMN REBOILER
DUTY - 780 M BTU/HR

2E-15
B₂ PRODUCT COLUMN REBOILER
DUTY - 100 M BTU/HR

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overhead from the B₅ concentrating column (2C-7) is cyclohexane-free pentaborane containing small percentages of C₅ and lighter hydrocarbons formed by the slight decomposition of cyclohexane in the pyrolysis reactor (2R-1). These are separated out in the B₅ product column (2C-8) and flared. The bottoms of this final column constitute the desired product, pure pentaborane-9.

Gas effluents from the tops of all but the B₅ product column (2C-8) are recovered by compressing (2K-2) and adding this process gas recycle stream to the reactor gas recycle stream. Liquid separated in the two knock-out drums associated with the compressor is pumped to the reactor phase separator (2PH-1).

3. Diborane Recovery from Hydrogen Off-Gas

As pointed out under the pyrolysis section, the reactor system is vented to the diborane absorber (2C-1) to handle the hydrogen generated by the pyrolysis reactions. This gas contains diborane in addition to the hydrogen and may also have small amounts of higher boranes. Actually this gas stream is three times as large as would result from the pyrolysis reactions alone, the greatest part being the consequence of the stripping gas used in the stripping columns (2C-2, 3, 4).

The quantity of cyclohexane recovered from the C₆ distillation column (2C-6), supplemented by the process make-up requirements, is insufficient to absorb the diborane contained in the gas fed to the diborane absorber (2C-1). The larger absorbent requirement, coupled with the need for considerable amounts of stripping gas, is satisfied by operating the absorbent stripper (2C-2) in conjunction with the diborane absorber (2C-1). This pair of columns, functioning at different pressure and temperature levels, produces a liquid stream, the bottoms of the stripper (2C-2), and a gas stream from the absorber (2C-1), both of which are relatively free of diborane and therefore suitable for use as absorbent and stripping gas, respectively.

However, the absorbent thus produced contains some intermediate boron hydrides which, if fed back to the absorber at the top plate, would result in boron losses in the hydrogen effluent. This situation is avoided by feeding the recycle absorbent several plates below the top plate of the absorber and feeding the fresh cyclohexane to the top of the column. Any intermediate boron hydrides rising from the recycle absorbent feed plate are absorbed in this short section of the absorber. In addition, the hydrogen gas loading on the top of the absorber is reduced by removing hydrogen used for stripping gas at a point intermediate between the top plate and the recirculating absorbent feed plate, since a high purity hydrogen stripping gas is not essential.

Gas effluent from the diborane absorber (2C-1) can be either recycled to Step I, the diborane plant, after removal of cyclohexane, or

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flared, depending on the cost of fresh hydrogen. The gas effluent from the absorber stripper (2C-2) joins the process gas recycle stream. The liquid effluent from the absorber is fed to the reactor phase separator (2PH-1) as make-up to the reactor system.

4. Analytical Instruments

Many of the equipment items are designed to produce certain compositions and the pyrolysis reactor (2R-1) is operated at a particular diborane concentration in the feed gas, achieved by adjusting the fresh diborane feed rate. Plant operators must therefore have pertinent analytical information quickly available.

This information is best provided in most instances through the use of automatic stream analyzers based on gas chromatography. All important gas streams are monitored at programmed intervals by this means. This method also is applied to certain liquid streams, e.g., the distillation column overheads, through analysis of the vapor existing in equilibrium with the liquid.

The two product streams are analyzed by other means, however. The 20 mole per cent solution of decaborane in cyclohexane is monitored by a process refractometer, and hydrocarbon impurities in the final pentaborane-9 stream by a continuous infrared analyzer.

B. Decaborane Production

The fundamental operating difference when decaborane is the desired product of the pyrolysis plant is that pentaborane-9 is recycled to the reactor system rather than being removed as a product. The reactor liquid in the phase separator (2PH-1) thus has a substantially higher pentaborane content (30 mole per cent vs. 4 mole per cent), and the reactor recycle gas carries back B_5H_9 to the reactor for conversion to decaborane.

Since the reactor liquid is already this concentrated, operation of the B_5 column (2C-5) is unnecessary. Instead this stream is fed directly to the B_5 concentrating column (2C-7), and the overhead from this is processed as before in the B_5 product column (2C-8) to remove light hydrocarbons. The bottom liquid from this final column, now containing 5 mole per cent cyclohexane, is returned to the phase separator (2PH-1).

Similarly operation of the C_6 column (2C-6) is also unnecessary because the bottoms liquid from the B_5 concentrating column (2C-7) has a high decaborane concentration. Therefore this stream is merely centrifuged to remove suspended solids, adjusted to the desired 20 per cent concentration with wash cyclohexane, and sent to storage (2C-11) to wait further processing to either crystalline decaborane or HEF-3.

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C. Decaborane Crystallization (Step IIA)

The pyrolysis section produces decaborane as a 20 mole per cent solution in cyclohexane. When the desired final product is HEF-3, this solution is sent to the alkylation plant, Step III; otherwise, the solution is processed to recover crystalline decaborane in Step IIA. This section of the plant is sized to accommodate the full output of the pyrolysis section, i.e., 9.2 tons per day. When pentaborane-9 is the primary product, however, only 0.6 ton per day of decaborane is produced. In this circumstance Step IIA is operated on an intermittent basis with the solution being accumulated in the storage tank (2C-11) between runs. The capacity of this tank is large enough to permit a 24 hour crystallizer run.

Figure 4 presents the process flowsheet for this operation, with related material balance information appearing in Table 4.

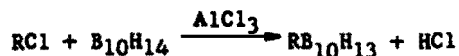
The decaborane solution is pumped (2G-12) from the holding tank (2C-11) to the decaborane concentrator (2C-12) where sufficient cyclohexane is evaporated to increase the concentration to 35 mole per cent. The cyclohexane overhead from this operation is returned to the make-up cyclohexane storage tank (2C-9) for reuse in the pyrolysis unit. The concentrated decaborane solution, after passing through a polishing filter, is continuously fed by pump (2G-12) to the crystallizer where cooling of the solution to 100°F. causes decaborane to crystallize out.

Slurry from the crystallizer flows by gravity to the centrifuge (2K-3). This unit automatically unloads into the steam heated rotary dryer (2D-1) through which hot nitrogen is circulated to remove residual cyclohexane. The dryer and centrifuge are kept only a few inches of water above atmospheric pressure and are further enclosed in a well ventilated housing in order to avoid atmospheric contamination with decaborane vapors. The dried product, after passing through a cooling section at the discharge of the dryer, is directly packaged in the final shipping container.

III. Decaborane Alkylation (Step III)

HEF-3 is produced by the liquid-gas reaction of decaborane and ethyl chloride, using dispersed aluminum chloride as a catalyst and cyclohexane as solvent. The alkylation unit is designed to use as feed the decaborane produced in the pyrolysis section (Step II). When the pyrolysis section is set up primarily for the production of pentaborane and only by-product amounts of decaborane are obtained, storage facilities allow for the accumulation of the decaborane solution until a sufficient quantity is available for operation of the alkylation section for a 24 hour period.

The alkylation of decaborane proceeds according to the following reaction:



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As in pyrolysis, several products can be formed including di-, tri- and poly-alkylated decaboranes. The percentage of multi-alkylated decaborane increases as the reactor conversion of decaborane is increased as indicated in Figure 5. Reactor conversions are therefore limited to 45 per cent to take advantage of maximum conversion to the mono-alkyl decaborane. Subsequent to the reaction, the HEF, consisting primarily of the mono- and di-substitutes, is separated from cyclohexane, unreacted decaborane and undesirable reaction products by a series of distillations. Because of the high boiling point of the product and the desirability of working at low temperatures to prevent product degradation, vacuum distillation techniques are employed. The process can be divided into three main steps, as follows:

- A. Alkylation
- B. Separation of Cyclohexane and Decaborane
- C. Purification of the HEF Product

The flowsheet for the process is presented as Figure 6 and the related material balance is provided by Table 5.

A. Alkylation

The feed stream coming from the pyrolysis section (Step II) contains 20 per cent decaborane in cyclohexane. It is mixed with the recycle decaborane stream of the same composition coming from the recycle concentrator (3C-1), and is fed to the alkylator feed tank (3C-3). The aluminum chloride catalyst is added to the feed tank (3C-3) which is agitated and heated to prevent catalyst settling and precipitation of the decaborane. The mixed catalyst and feed are fed by gravity and pressure difference to the alkylation reactor (3R-1).

A multi-stage liquid gas contact reactor is required and, since the alkylating agent is a gas, the reactor consists of a jacketed, bubble cap tower in which the gas velocity is slightly higher than normal to maintain suspension of the aluminum chloride. Ethyl chloride is fed into the bottom of the reactor and flows countercurrently to the liquid. As the reaction proceeds, ethyl chloride is replaced mole for mole by hydrogen chloride, resulting in a constant molal gas flow. The ethyl chloride flowing up the column has the additional effect of stripping hydrogen chloride from the reactor effluent. A reactor hold-up time of 2 hours is used during which time all of the ethyl chloride is reacted.

The off-gas from the reactor (3R-1) consists essentially of HCl with a small amount of C_6H_{12} and is disposed of by scrubbing with water. If economies warrant, both these chemicals can be separated, purified and recycled back to an earlier stage of the integrated plant process.

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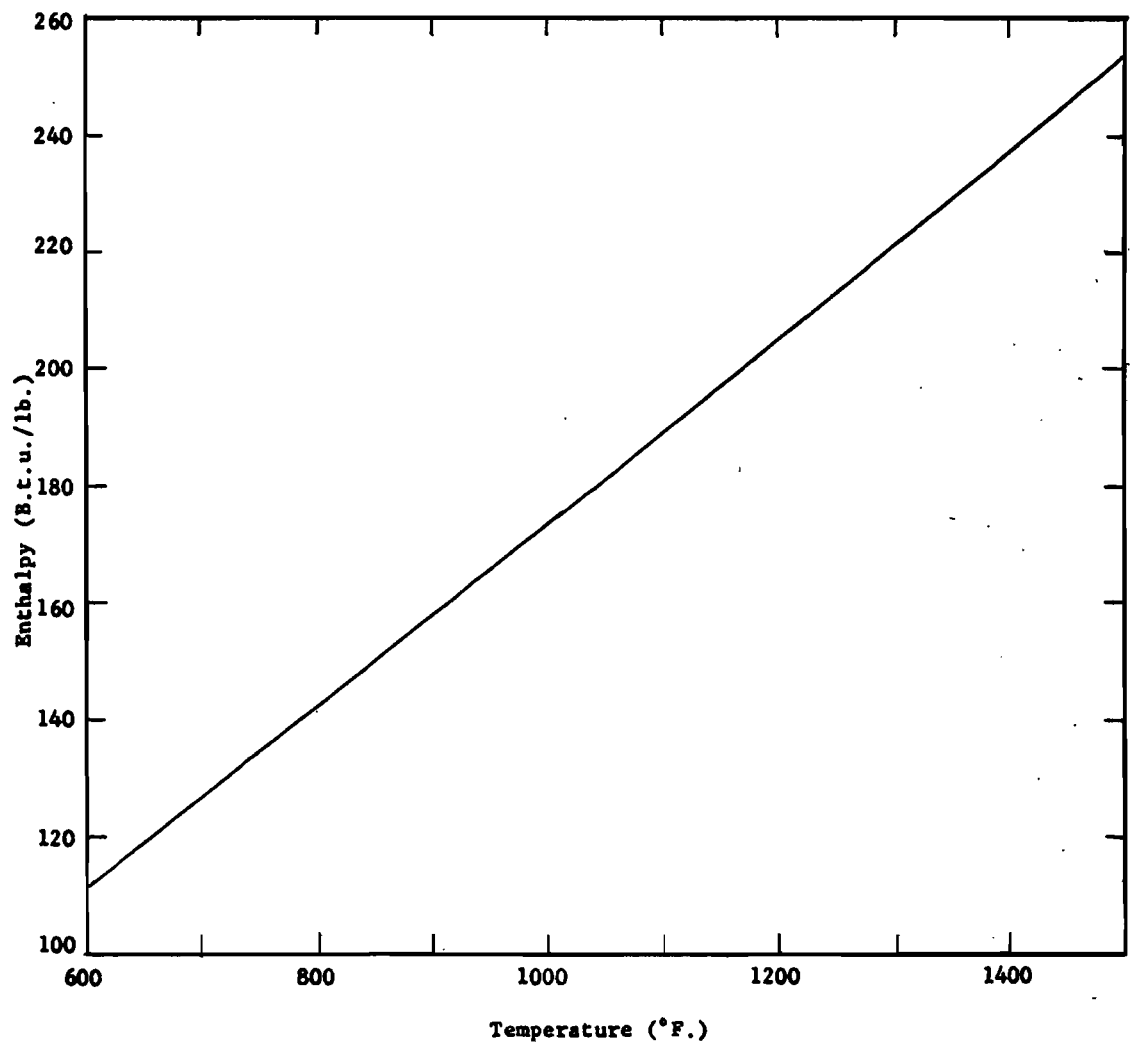


Figure B-8. Enthalpy of BCl_3 (high range)

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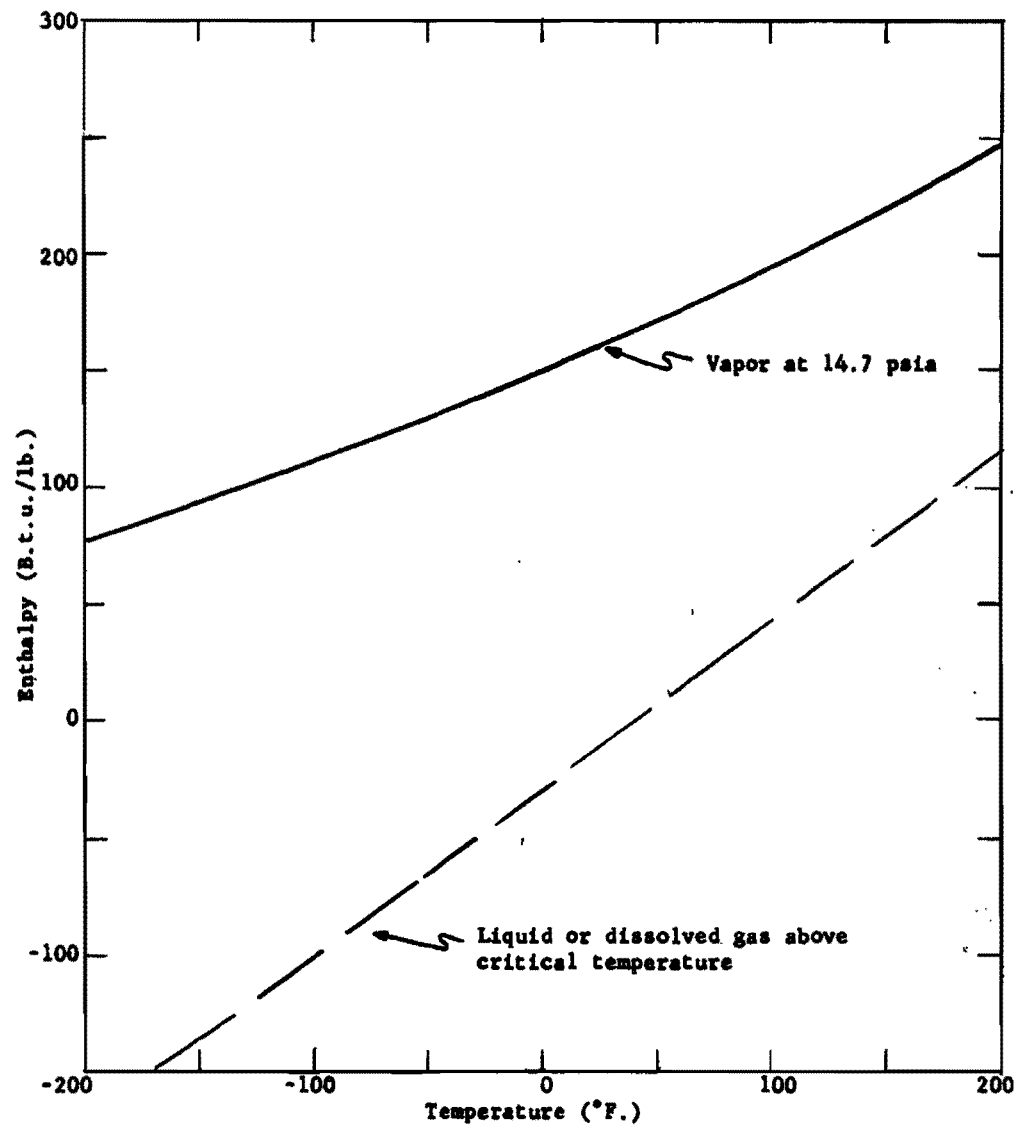


Figure B-9. Enthalpy of Diborane

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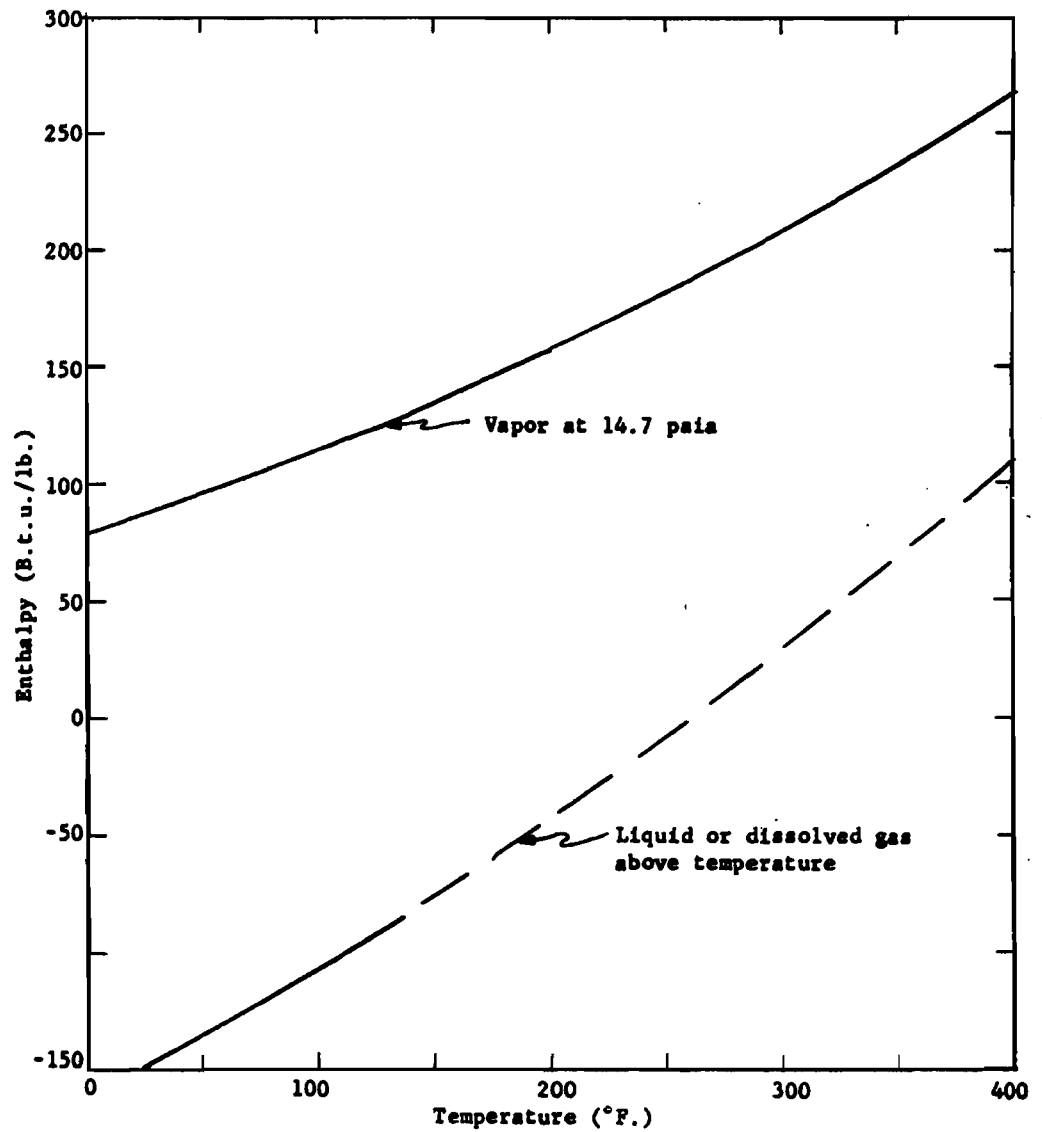


Figure B-10. Enthalpy of Pentaborane, B_5H_9 and B_5H_{11}

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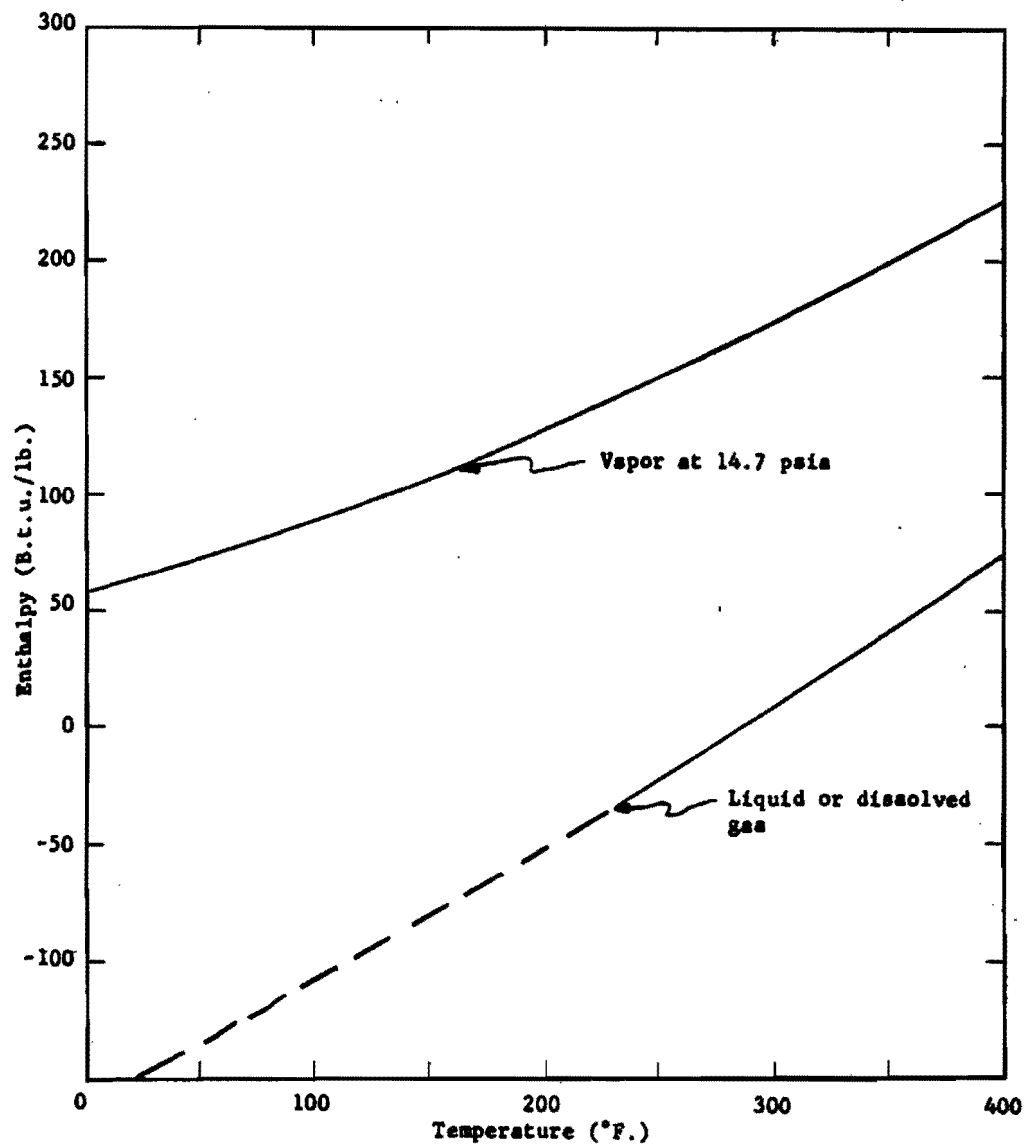


Figure B-11. Enthalpy of Decaborane

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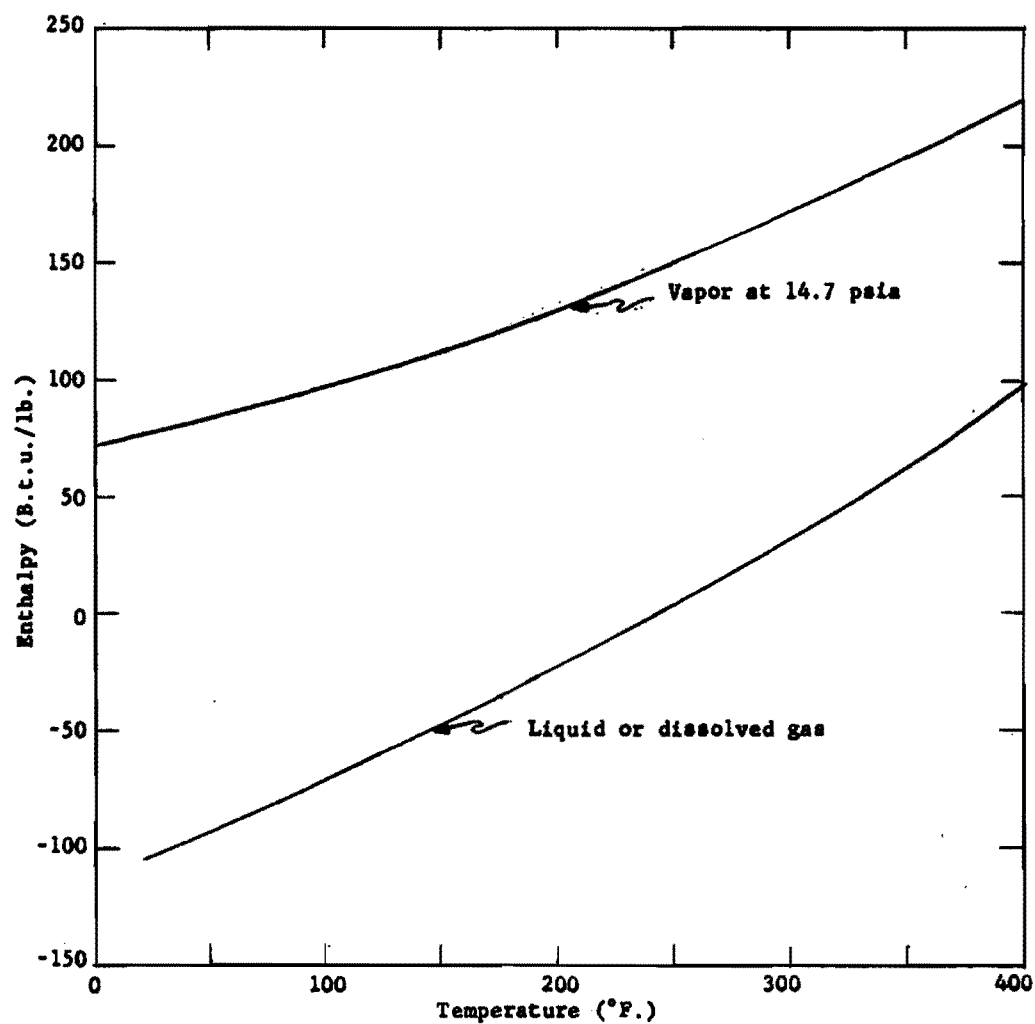


Figure B-12. Enthalpy of Cyclohexane

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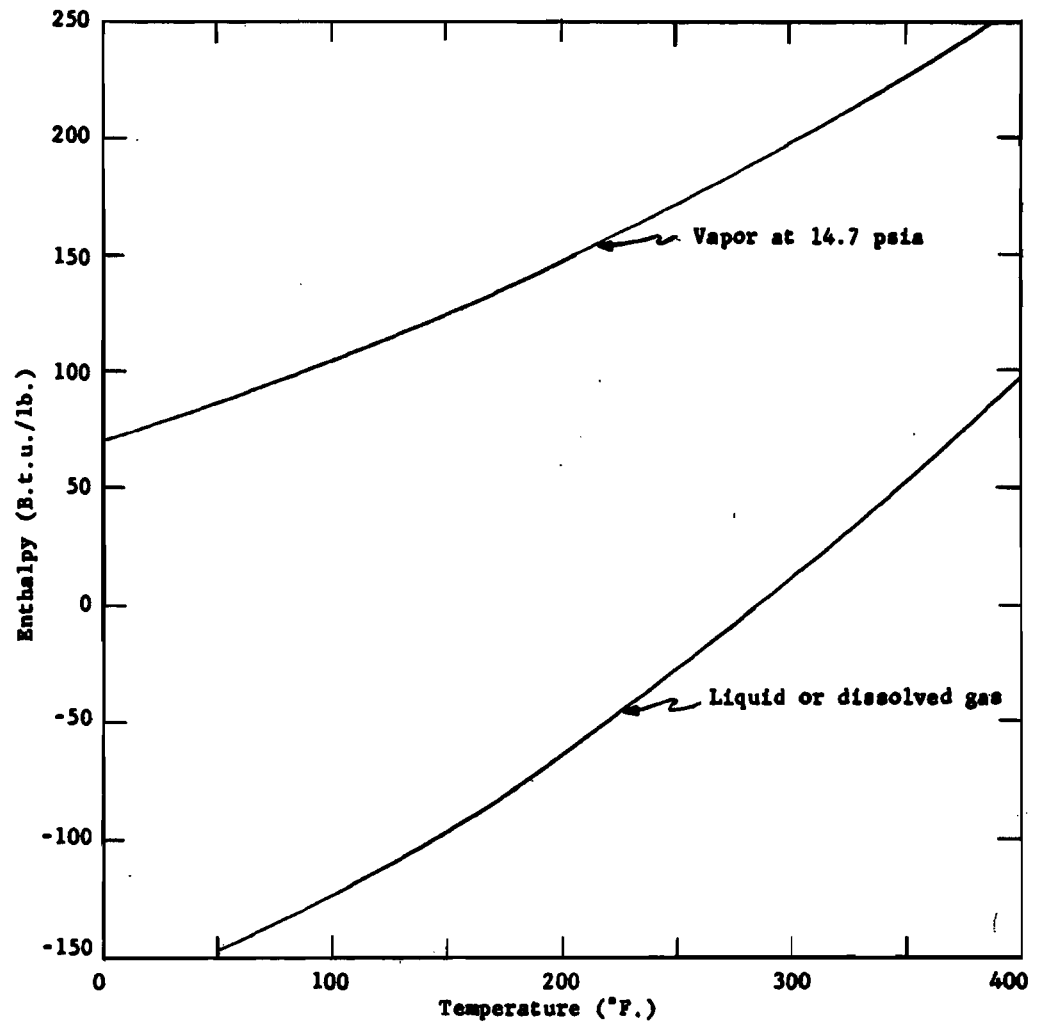


Figure B-13. Enthalpy of Hexaborane

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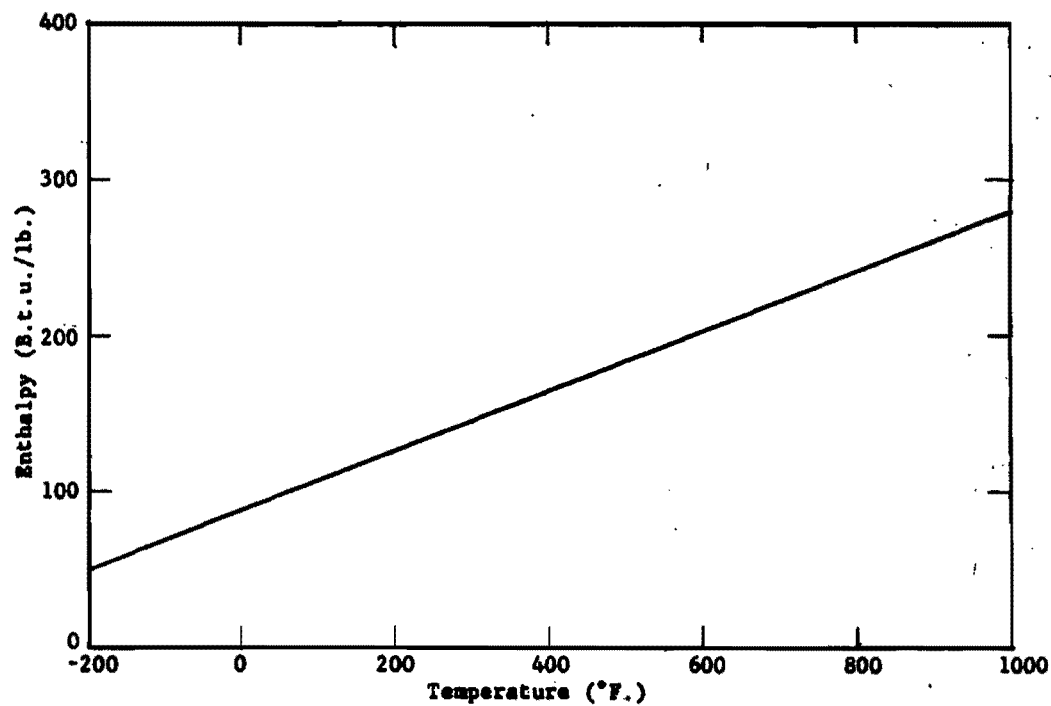


Figure B-14. Vapor Enthalpy of HCl (anhydrous)

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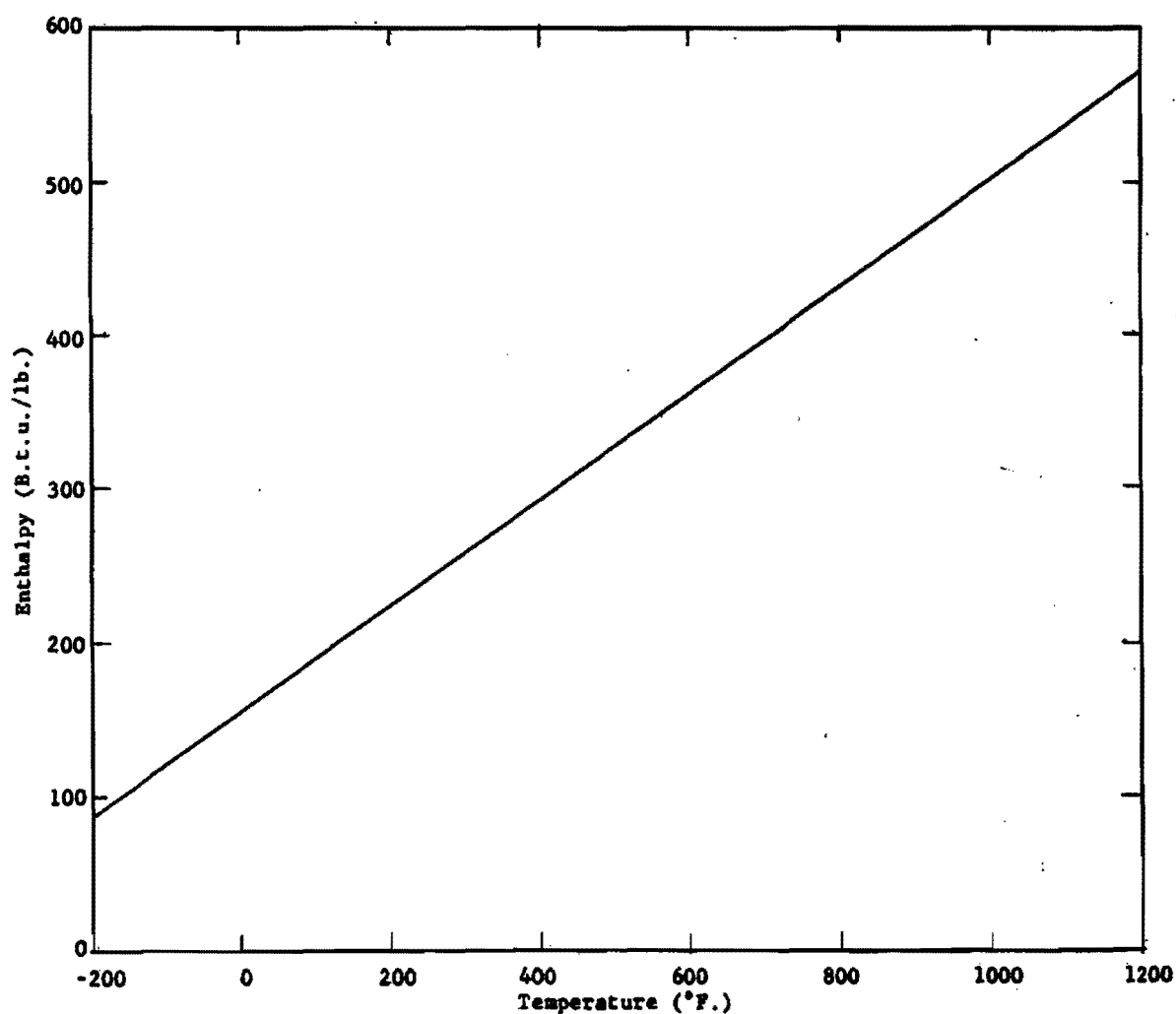


Figure B-15. Vapor Enthalpy of Hydrogen

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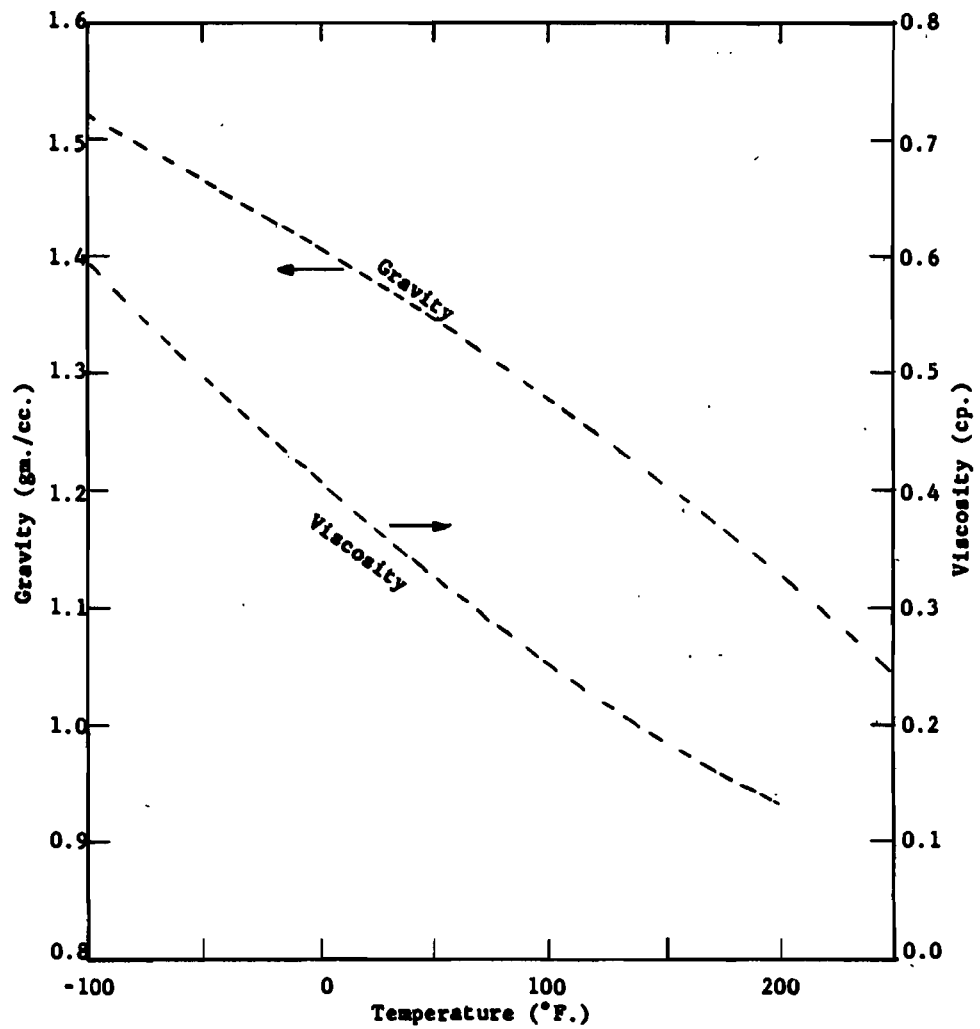


Figure B-16. Gravity and Viscosity of BCl₃

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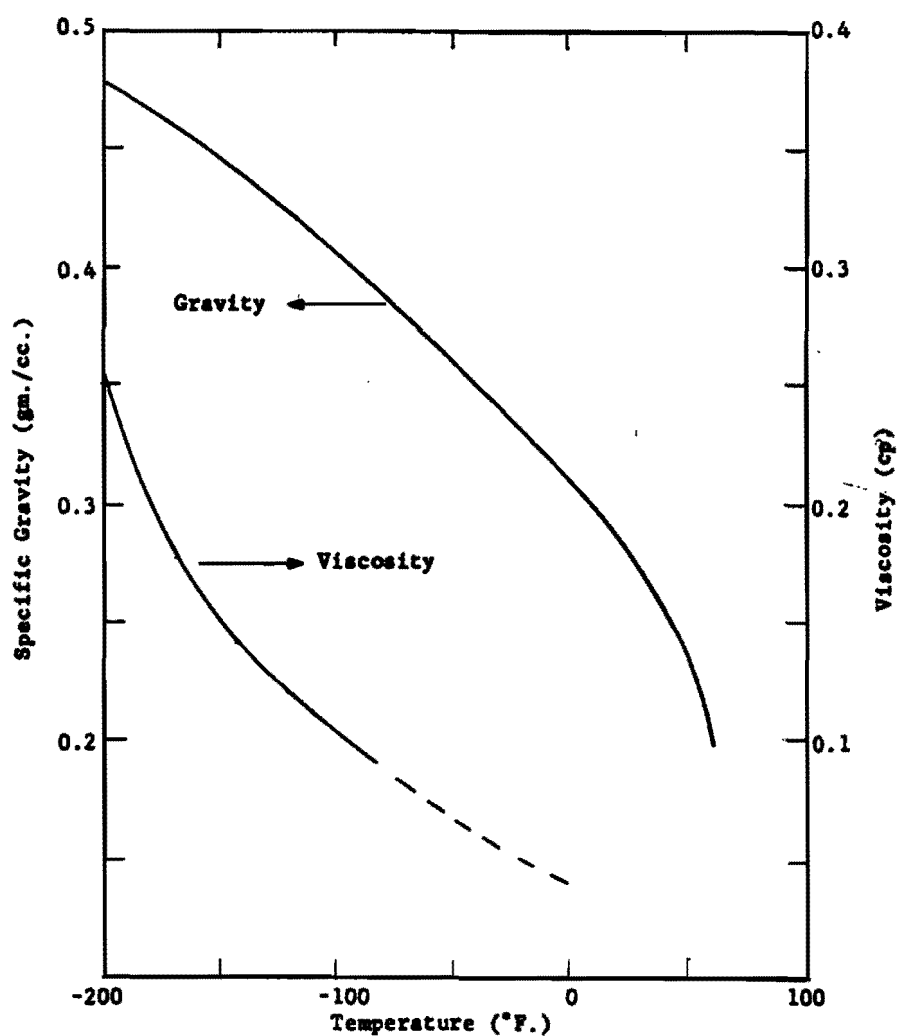


Figure B-17. Viscosity and Specific Gravity of Diborane (liquid)

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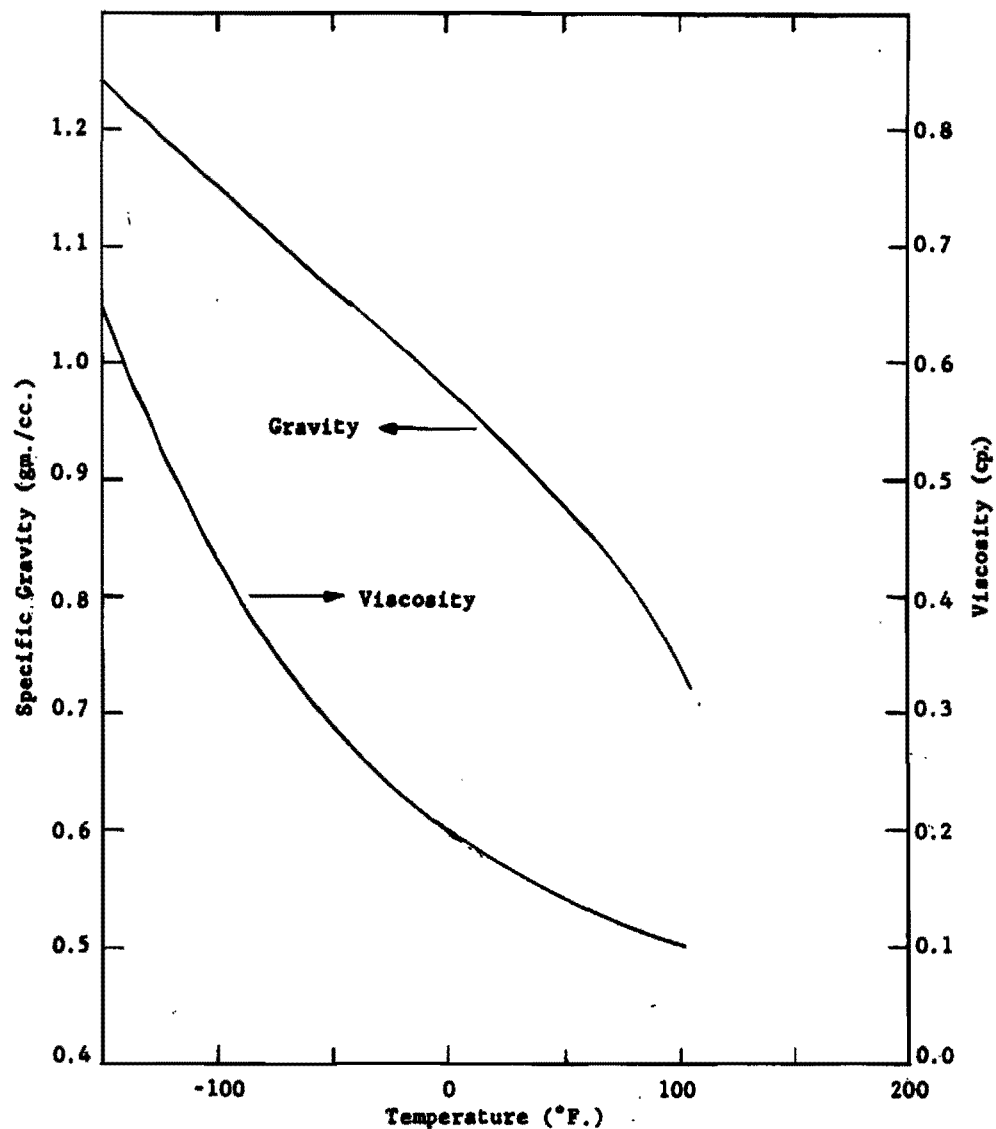


Figure B-18. Estimated Gravity and Viscosity of Anhydrous HCL (liquid)

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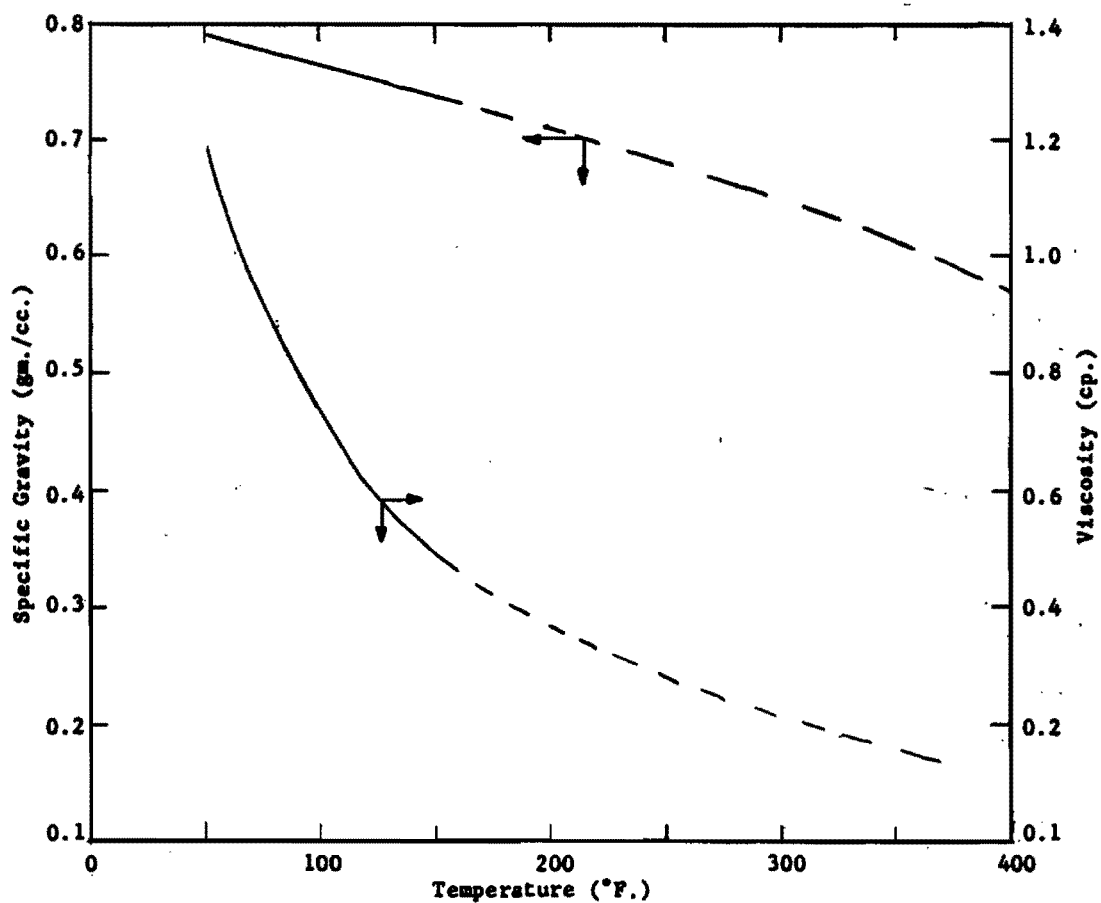


Figure B-19. Viscosity and Specific Gravity of Cyclohexane Liquid

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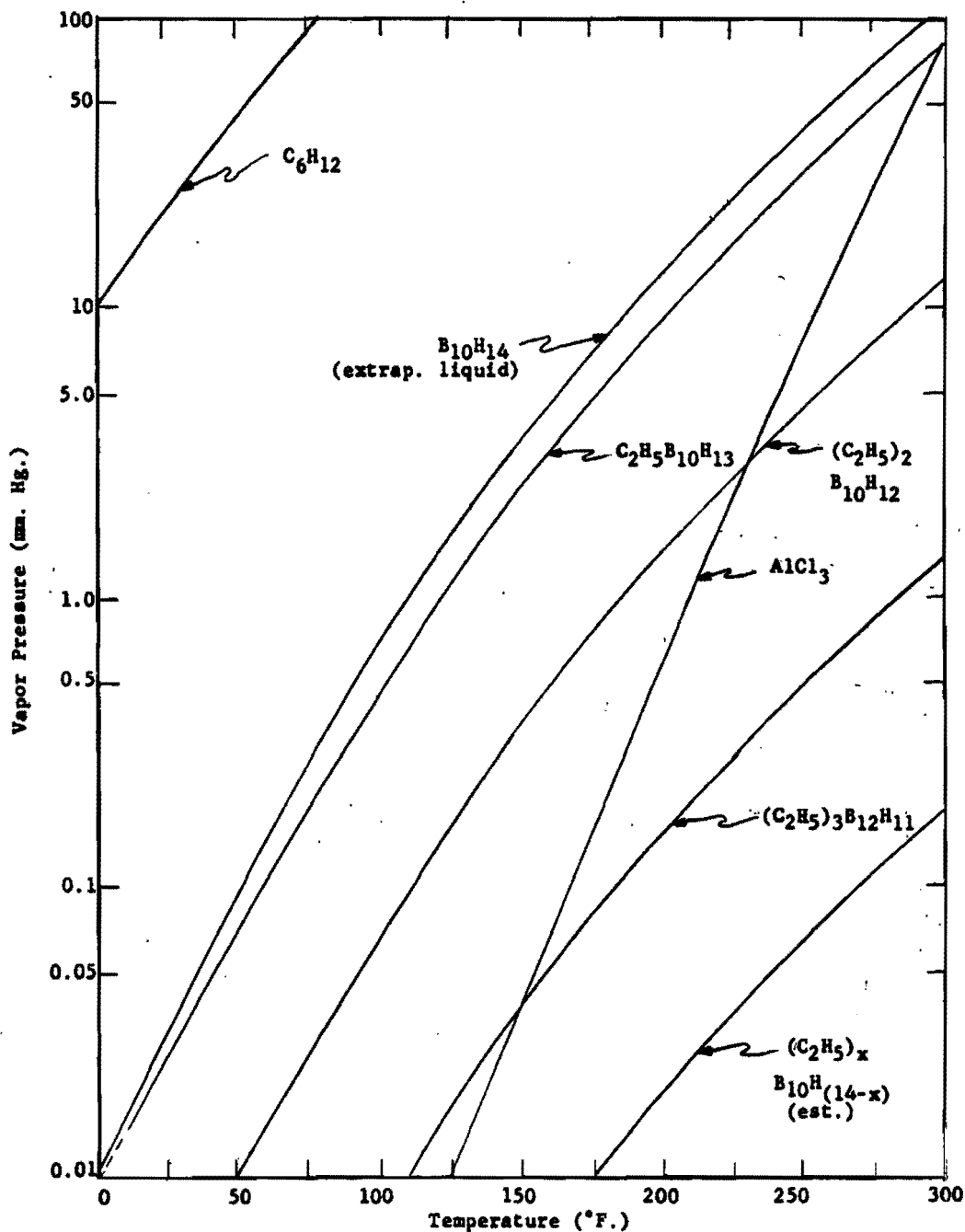


Figure B-20. Vapor Pressures

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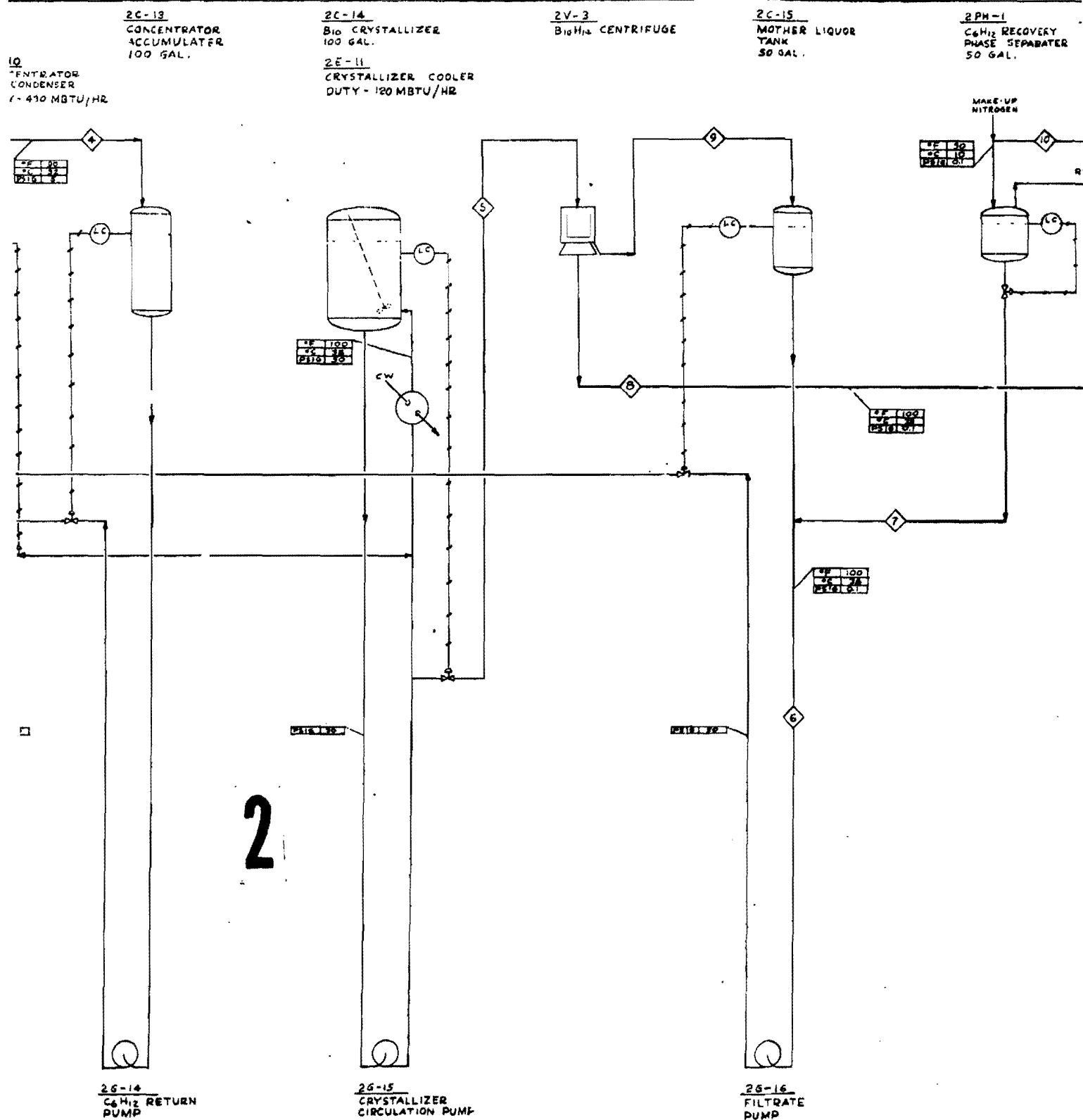
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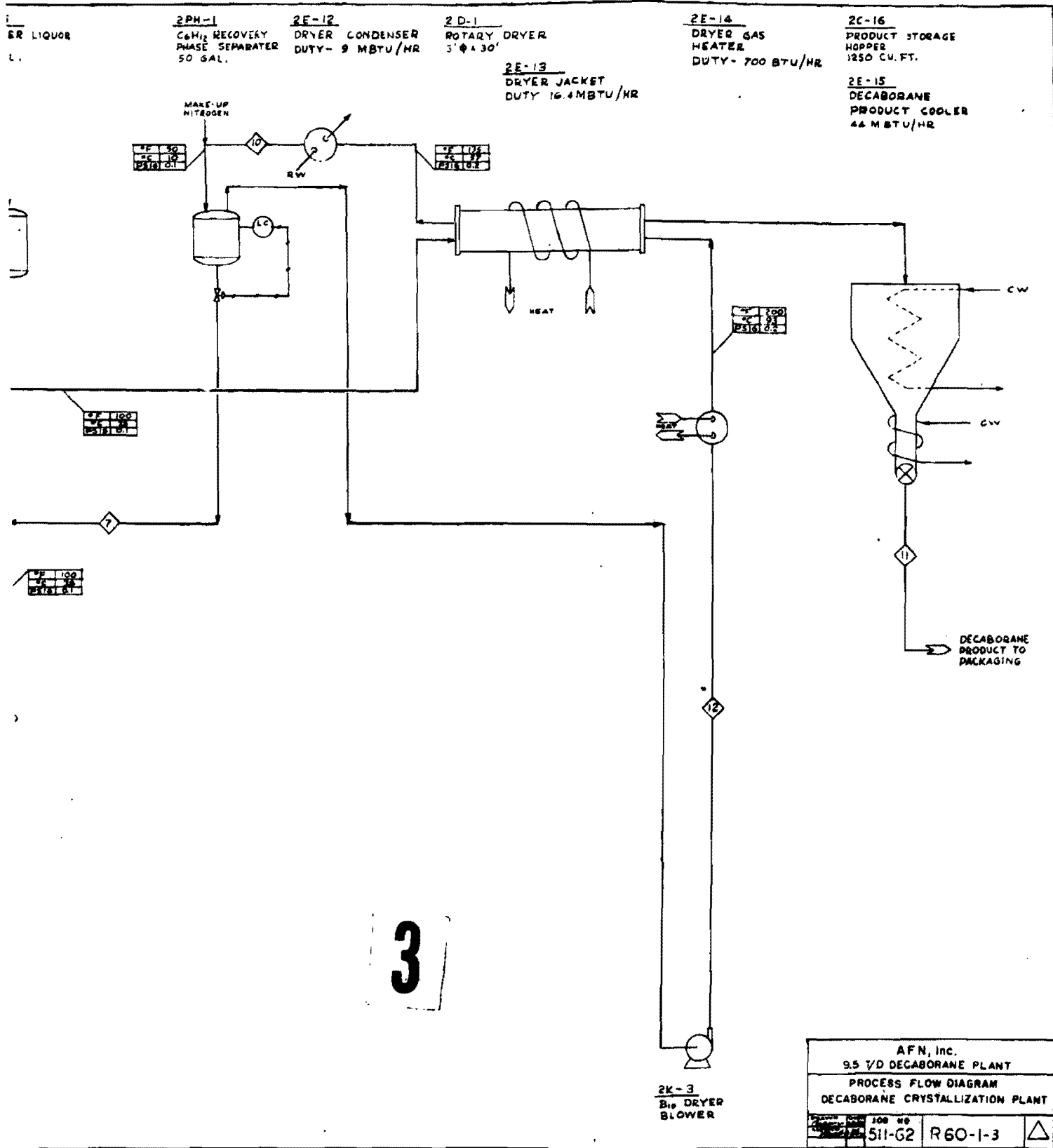
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TABLE 4

Decaborane Crystallization (Step IIA) Material Balance
(moles/hour)

Stream	Stream No.	B ₁₀ H ₁₄	N ₂	C ₆ H ₁₂	Total
Crystallization Section Feed	1	6.26		25.04	31.30
Concentrator Feed	2	7.89		35.06	42.95
Concentrator Overhead	3	0.34		25.04	25.38
Crystallizer Feed	4	7.55		10.02	17.57
Crystallizer Slurry	5	7.55		10.02	17.57
Concentrator Recycle	6	1.63		10.02	11.65
Drier Condensate	7			0.70	0.70
Centrifuge Solids	8	5.92		0.70	6.62
Centrifuge Mother Liquor	9	1.63		9.32	10.95
Drier Off-Gas	10		2.14	0.80	2.94
Decaborane Product	11	5.92			5.92
Drying Gas	12		2.14	0.10	2.24

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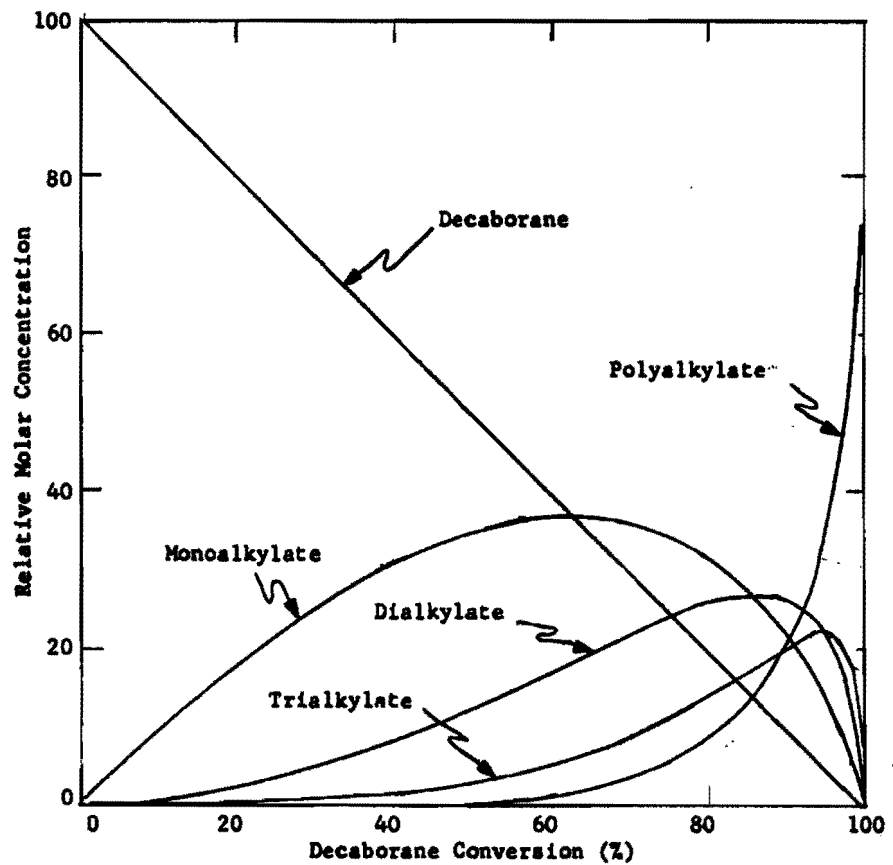
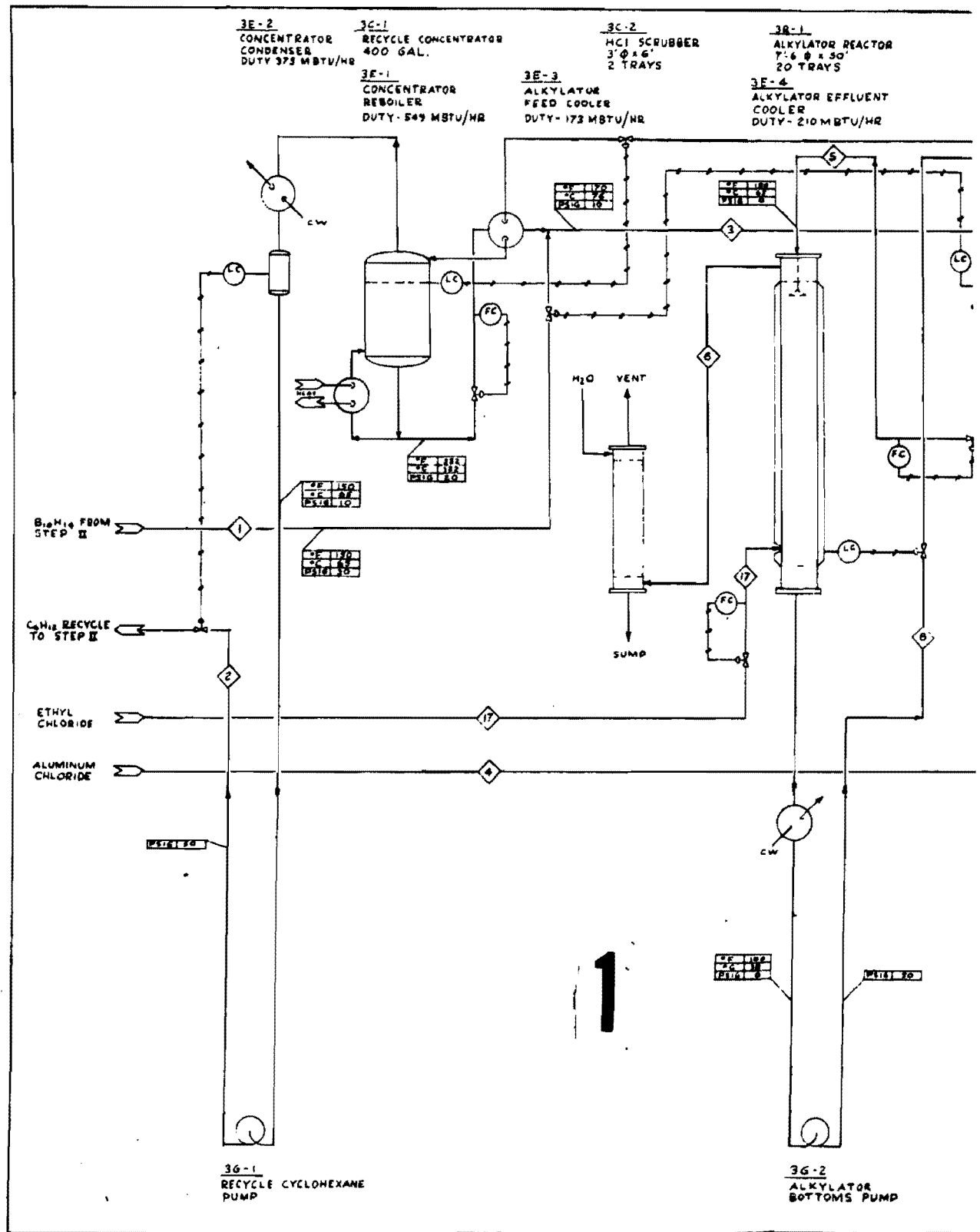


Figure 5. Distribution of Compounds in Alkylation Mixture

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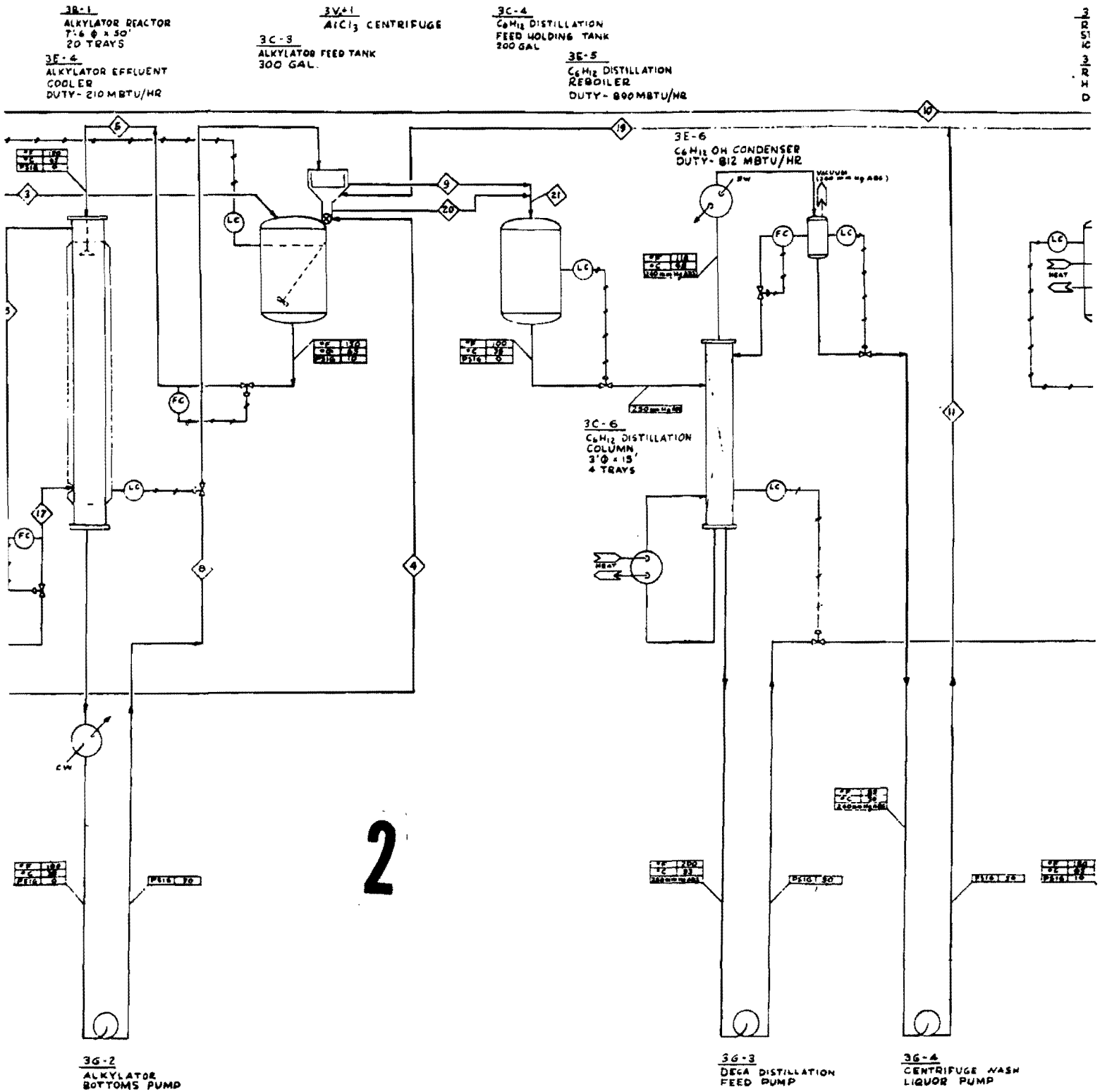
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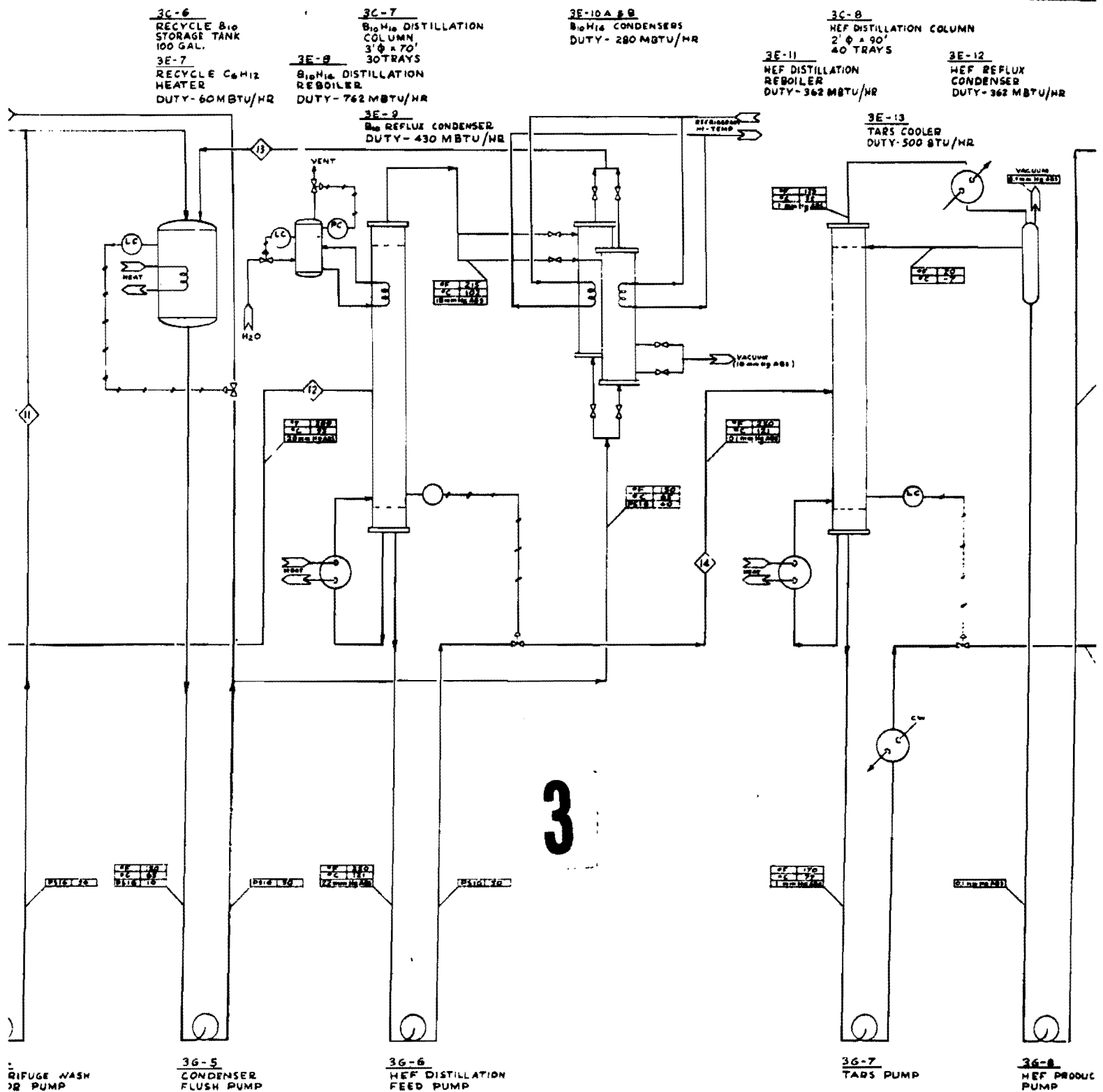
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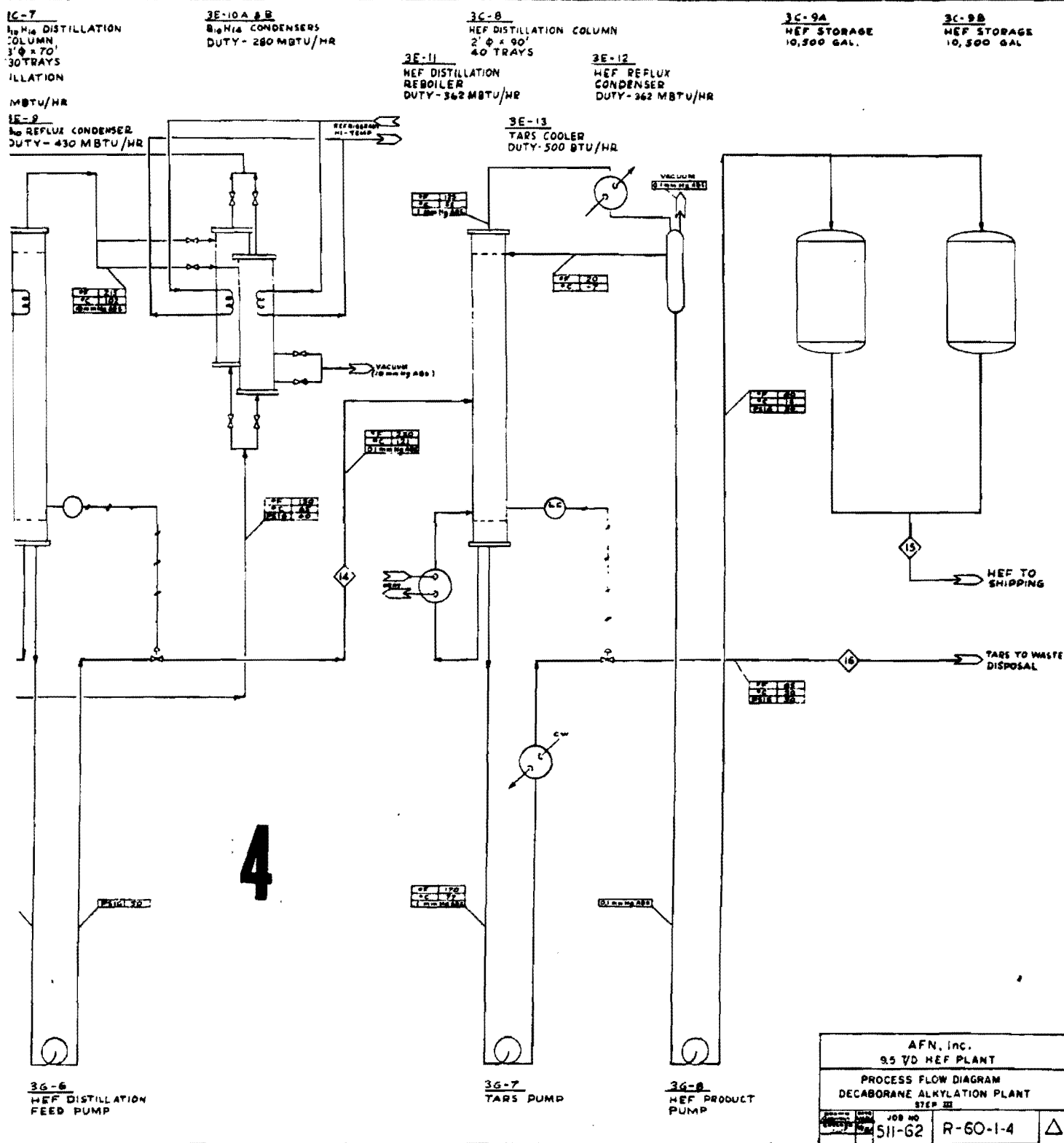


TABLE 5

Decaborane Alkylation (Step III) Material Balance
(moles/hour)

Stream	Stream No.	C ₆ H ₁₂	B ₁₀ H ₁₄	Mono-ethyl B ₁₀ H ₁₄	Di-ethyl B ₁₀ H ₁₄	Tri-ethyl B ₁₀ H ₁₄	Poly-ethyl B ₁₀ H ₁₄	AlCl ₃	Ethyl Chloride	HCl	Total
Feed to Alky. Section from Step II	1	25.080	6.270	-	-	-	-	-	-	-	31.350
C ₆ H ₁₂ Take-Off	2	24.754	0.001	-	-	-	-	-	-	-	24.655
Feed to Alkylator	3	30.217	13.489	0.091	-	-	-	-	-	-	43.797
AlCl ₃ Make-Up	4	-	-	-	-	-	-	0.044	-	-	0.044
Catalyzed Feed to Alkylator	5	31.464	13.582	0.158	0.016	0.003	0.001	2.736	-	-	47.960
Alkylator Off-Gas	6	0.326	-	-	-	-	-	-	-	7.864	8.190
Centrifuge Solids	7	1.573	0.093	0.067	0.016	0.003	0.001	2.736	-	-	4.489
Alkylator Effluent	8	31.464	7.470	4.613	1.310	0.270	0.007	2.736	-	-	47.870
Centrifuge Liquor	9	29.891	7.097	4.344	1.244	0.256	0.006	0.037	-	-	42.875
Recycle C ₆ H ₁₂	10	29.891	7.221	0.091	-	-	-	-	-	-	37.203
C ₆ H ₁₂ Dist. Overhead	11	31.430	0.007	-	-	-	-	-	-	-	31.437
C ₆ H ₁₂ Dist. Bottoms	12	3.185	7.371	4.546	1.293	0.267	0.007	0.044	-	-	16.713
B ₁₀ H ₁₄ Dist. Overhead	13	3.185	7.215	0.091	-	-	-	-	-	-	10.491
Feed to HEF Dist. Column	14	-	0.155	4.455	1.293	0.267	0.007	0.044	-	-	6.221
Final Product	15	-	0.155	4.455	1.292	0.261	0.001	-	-	-	6.164
HEF Dist. Residue	16	-	-	-	-	0.006	0.006	0.044	-	-	0.056
Ethyl Chloride Feed	17	-	-	-	-	-	-	-	7.864	-	7.864
Alkylator Recycle	18	26.706	0.006	-	-	-	-	-	-	-	26.712
Centrifuge Solids Wash Liquor	19	4.724	0.001	-	-	-	-	-	-	-	4.725
Recycle Solids Wash Liquor	20	4.724	0.281	0.202	0.049	0.010	0.001	0.007	-	-	5.274
Feed to C ₆ H ₁₂ Dist. Col.	21	34.615	7.378	4.546	1.293	0.267	0.007	0.044	-	-	48.150

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B. Separation of Cyclohexane and Decaborane

The reactor effluent is cooled (3E-4) to lessen the solubility of aluminum chloride and pumped (3G-2) to a centrifuge (3V-1). The solid aluminum chloride is removed, washed and recycled to the alkylator feed tank (3C-3) while the mother liquor, together with the wash liquor, is fed to the C_6H_{12} distillation feed holding tank (3C-4) and then to the C_6H_{12} distillation column (3C-5). Cyclohexane is removed under vacuum from the product stream, condensed (3E-6) and pumped (3G-4) to the recycle storage tank (3C-6) where it is used as a wash and diluent for the decaborane condensers (3E-10A and B).

To minimize decomposition, the bottoms temperature of the C_6H_{12} distillation column (3C-5) is limited to 200°F. The bottoms containing cyclohexane, decaborane and the alkyl substituted decaboranes, are pumped (3G-3) to the decaborane distillation column (3C-8).

Again, to limit the column temperatures, vacuum distillation techniques are employed. Decaborane is refluxed by using an internal, partial condenser while cyclohexane acts as a non-condensable. The overhead decaborane-cyclohexane line is steam traced to prevent the premature condensation of solids prior to the introduction of the streams into the condensers (3C-10A and B). Decaborane and cyclohexane are collected as solids in the refrigerated condensers (3C-10A and B) which are alternately used and washed with warm cyclohexane from the recycle storage tank (3C-6). This material is circulated back to the recycle storage tank (3C-6) from which it is pumped (3G-5) to the recycle concentrator (3C-1) and then fed to the alkylator (3R-1).

C. HEF Purification

Crude HEF coming from the bottom of the decaborane distillation column (3C-7) contains small amounts of polyalkylated decaborane tars and dissolved aluminum chloride. In order to remove these contaminants, the $B_{10}H_{14}$ distillation column (3C-7) bottoms are pumped (3G-6) to the HEF distillation column (3C-8) for final purification. The overhead product stream is monitored using process refractometers, in addition to normal laboratory analytical coverage. The purified HEF, consisting primarily of mon-alkyl decaborane with minor amounts of decaborane and higher alkylated boranes, is pumped (3G-8) to the HEF storage tanks (3C-10) and kept under a nitrogen atmosphere. The tars coming from the column bottom are pumped (3G-7) to the waste disposal area where they are incinerated.

IV. Equipment Design

All major equipment items are listed in Appendix A along with specific sizing data.

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A. Reactors

1. Hydrogenation Reactor (Step I)

The hydrogenation preheater and reactor (1F-1 and 1F-2) are combined into a single unit because of the similarity in operating conditions. The reactor is designed to hydrogenate boron trichloride at a temperature of 1275°F. with a retention time of 0.5 second. The required throughput is 7000 cu.ft./min. and reactor volume is therefore 79 cubic feet.

The preheater and reactor consist of four parallel helical coils in a gas-fired refractory furnace. Each coil is constructed of 6 inch I.D. stainless steel tubing with a one inch wall. The coils are 230 feet long, of which 125 feet are preheater and 105 feet are reactor. The reactor section is packed with No. 20 mesh silver wire screen and because of the severe corrosion problems, the reactor and preheater are both lined with 1/16 inch silver.

Temperature sensing elements mounted on the tube walls protect against overheating and sloughing of the silver lining.

2. Pyrolysis Reactor (Step II)

The diborane pyrolysis reactor (2R-1) is designed to pyrolyze diborane with a 2 second retention time at a 10,500 cfm recirculation rate at 500°F. Total required reactor volume for this condition is 340 cubic feet. Material of construction is carbon steel. Because of clean-out requirements, three reactor units each with 170 cubic feet of volume are provided. This permits operation of two units while a third one is being cleaned and placed in standby condition. Each unit consists of five 18 inch diameter reactor tubes 21 feet long, individually jacketed, with a cyclohexane quench crossmounted on each tube. Underneath each tube, a 3 foot long scouring rod is mounted on a hydraulic cylinder to clean out the quench cross and bottom section of reactor tube. The cyclohexane quench velocity matches the gas velocity at the tube exit to eliminate pressure drop. The scouring rod keeps the quench nozzle clean and free. The hydraulic rams are activated whenever pressure drop across any reactor tube increases above a set maximum. Reactor heating is accomplished by a gas-fired Aroclor heating system.

3. Alkylation Reactor (Step III)

The alkylation reactor (3R-1) is designed for the catalytic alkylation of decaborane at atmospheric pressure, 150°F., and a 2 hour retention time. The required throughput is 550 gallons per hour decaborane solution and 470 cubic feet per hour alkylating agent.

The reactor consists of a hot water-jacketed, bubble cap tower in which the required multi-stage reaction is accomplished by countercurrently

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feeding the catalyzed decaborane solution and the gaseous ethyl chloride. Adiabatic conditions are maintained throughout the reaction vessel. Because of the suspended aluminum chloride catalyst the gas rates through the bubble caps are maintained at a slightly higher than normal rate to prevent settling.

The reactor is 7.5 feet in diameter by 50 feet high and contains 20 trays; it is constructed of carbon steel.

B. Columns and Vessels

All columns having a diameter greater than two feet are of standard bubble cap design while those under two feet are packed columns using one inch Intalox saddles. The high vacuum columns used in the alkylation section have a maximum allowable pressure drop of 0.1 mm. Hg. and are therefore of the low pressure drop mechanical type.

Columns and vessels in service below -40°F. are fabricated of type 304 stainless steel to provide low temperature impact resistance. All other columns and vessels are carbon steel except the final HEP-3 distillation column (3C-8) which is also type 304 stainless steel.

Storage tanks which require either auxiliary heating or cooling are fabricated with integral helical coil heat exchangers except where external jackets are specified.

C. Heat Exchangers

Heat exchangers will generally be of the shell and tube type except in isolated cases where concentric tube, internal helical coils or jacketed vessels are specified.

Heat exchangers in service below -40°F. are constructed of type 304 stainless steel to prevent failure due to embrittlement. Other exchangers are fabricated from carbon steel. The Step I reactor effluent cooler (1E-1) is lined with 1/16 inch silver on both sides of the tube and on the inner shell surfaces because of the severe corrosive conditions under which it operates.

The reactors discussed earlier in each case also act as heat exchangers and are designed and fabricated accordingly.

D. Pumps and Compressors

All pumps and compressors are spared. In the case of the Step II reactor recycle compressor (2K-1), one spare is used for two compressors recycling reactor gas. Where possible, pumps and compressors of similar capacities will be made identical to reduce parts inventory. Because of small amounts of boric oxide solids in Step I and boron hydride polymer in

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Step II, pumps and compressors using check valves are to be avoided. Polishing filters are used to control these solids; however, experience has shown that they cannot be completely eliminated without elaborate filtering equipment.

Pumps using tungsten carbide against carbon seal rings are recommended throughout the plant except for those pumping diborane-rich or pentaborane-rich streams. Step I pumps (1G-1 through 1G-5) would use single mechanical seals with a liquid boron trichloride flush stream. Step II pumps would use double mechanical seals with cyclohexane as the sealant.

Pumps handling diborane or pentaborane streams would be canned pumps to eliminate possible toxic or fire hazards caused by leakage. In cases where these flows are 3 gpm or less, diaphragm pumps are suggested.

Compressors would all be of the rotary type, either centrifugal or positive displacement with double mechanical seals on the shafts. Boron trichloride would be used in Step I and cyclohexane in Step II for the sealant fluid.

E. Filters and Centrifuges

Polishing filters used for process streams will be of vertical leaf design with mounted spares for simplicity in cleaning. Since only small amounts of solids will be accumulated, they can be destroyed instead of recycled, which simplifies filter cleaning. The cleaning procedure to be followed will be a hot caustic back wash after purging equipment, followed by rinsing and drying under vacuum. All polishing filters will be steam jacketed for ease in removing moisture.

Centrifuges to be used in Step II for removing boron hydride polymer will be of the pressurized automatic solids discharge type with a split bowl design. The centrifuge used for decaborane separation would be of the piston-operated solids discharge type. The polymer centrifuges have integrally mounted centrifugal pumps to remove filtrate which eliminates the necessity of externally mounted pumps. Where streams are large, two centrifuges will be mounted in parallel to even out flow with each centrifuge sized to handle the complete stream alone in case of failure of one.

F. Piping and Valves

Carbon steel tubing and piping will be used throughout the plant for process piping except where low temperature conditions (-40°F. or lower) exist. For low temperature service, type 304 stainless steel will be used for process piping. Carbon steel, 0.065 inch wall, annealed, seamless tubing will be used in only three sizes: 3/8 inch, 5/8 inch and 7/8 inch with carbon steel, cadmium-plated Imperial Hi-Seal fittings. Carbon steel Schedule 40 seamless Grade A/B ASTM A-53 pipe will be used for one inch and larger sizes with butt weld fittings. Flanges will be used only where required for equipment removal. Any screwed unions, tees, or elbows will be 3000 pound cast steel fittings.

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Stainless steel 0.049 inch wall, seamless, type 304 with type 316 stainless steel Imperial Hi-Seal fittings will be used for refrigerated process tubing in three sizes: 3/8 inch, 5/8 inch and 7/8 inch. Type 304 stainless steel Schedule 10S seamless pipe will be used with Schedule 10S butt welding type 304 stainless fittings in one inch to six inch sizes. Any required stainless screwed fittings will be 2000 pound type 304 stainless.

The valves used in process lines will be ball valves with Teflon seats and seals. For globe valve use, screwed steel body, union bonnet valves are used. Stainless steel, spring-loaded valves are to be used for check valve service in process lines. Welding neck flanges with 1/16 inch raised face will be specified for 300 pound and 150 pound service for use with process piping.

For refrigerant lines, hard drawn copper tubing, ASTM B-88, Type L will be used with brass valves and fittings. Refrigerant lines connected to threaded connections at equipment will be installed using a silver alloy, back-brazed copper adapter.

For instrument air, nitrogen and breathing air lines, Schedule 40 galvanized, ASTM A-120 pipe and galvanized fittings will be used. Copper tubing, 1/4 inch with 0.030 inch wall and 12 tube bundles of Dekron Protecto Pac Type 8 will be used for instrument air lines less than 1/2 inch in size.

Other utilities, cooling water, plant water, steam, vacuum, and chilled water under 125 psig and 350°F. will be Schedule 40 pipe ASTM A-53 Grade A/B with 150 pound brass gate, globe and check valves. Fittings will be malleable iron 150 pound. High pressure steam, 175 psig, and natural gas will use 3000 pound fittings, 600 pound valves of forged steel, Schedule 80 pipe for under one inch line size, and Schedule 40 pipe for over one inch line size.

In addition, process piping will be sloped 1/12 inch per foot in the direction of flow for draining. Teflon paste will be used on stainless threaded connections, Garlock 101 in Step I, and John Crane Plastic Lead Seal in Steps II and III for carbon steel process piping threaded connections as pipe thread compounds.

G. Instrumentation

Conventional flow, level and temperature instrumentation, either pneumatic or electronic, can be used throughout the plant. Materials of construction for the primary sensing elements, however, must be restricted to those compatible with boron hydrides and boron trichloride. In the diborane generation step where boron trichloride is present, Teflon is the only recommended polymeric material. In the remainder of the plant, Viton A and Teflon are satisfactory materials except in Step III equipment where hydrogen is generated. Here Teflon again is recommended. Steel or stainless

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steel are suggested except at the temperatures encountered in the Step I reactor and feed preheater where silver-lined primary elements are recommended in contact with process gas.

Process stream analyzers will be required throughout the plant to control and observe changes in the various processes. Applications for these are discussed in the process description sections of this volume.

V. Utilities

Utility requirements for the various process steps are presented in Table 6. Power requirements as given in the table include only process pump and compressor loads along with instrumentation and lighting loads for each step. Refrigeration, cooling tower water, chilled water, compressed air and vacuum power requirements are not included as part of the process power requirements. Power requirements in Step I include power for a 1900 h.p. drive on the hydrogen recycle compressor. Economic studies of a turbine steam drive versus an electrical drive may, depending on utility costs and steam plant size, eliminate this large electrical power requirement for Step I.

Compressed air requirements are based on pneumatic type instrumentation. If electrically operated instruments predominate, a lower compressed air requirement and proportionately larger electrical requirement will result.

As a safety precaution, fittings for breathing air (20°F. dewpoint), nitrogen, vacuum, water and steam will be of different types to prevent cross-connecting utility services.

VI. Waste Disposal

Waste material in the plant will consist mainly of process waste gas, process solids, and alkylation tars.

A. Vented Gases

A caustic scrubber normally containing 5000 gallons of 5 per cent caustic solution is used to remove vaporized boron hydrides, hydrogen chloride, and boron trichloride which accompany hydrogen process off-gases from the diborane plant and alkylation plant. All rupture discs and safety relief valves in the process vessels of the plant release their vapors to this scrubber in case of pressure build-up. To prevent back inspiration of air into the system during normal operation, nitrogen is bled into the ends of the gas manifold. To prevent air inspiration after a rupture disc or safety relief valve has opened, a gas pressure control valve releases nitrogen into the manifold system when manifold pressure drops below four inches of water above atmospheric pressure. A caustic solution pump circulates

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TABLE 6

Process Utility Requirements

Utility	Step I Diborane Production	Step II Diborane Pyrolysis	Step IIA B ₁₀ H ₁₄ Crystallization	Step III HEP-3	Total
<u>Steam</u>					
40 psig, lb./hr.	87,920	23,000	370	2,630	113,920
150 psig, lb./hr.	22,540	7,000	-	-	29,540
<u>Cooling Tower Water</u>					
60 psig, 80°F., gpm	4,790	2,340	60	90	7,280
<u>Chilled Water</u>					
60 psig, 40°F., gpm	-	115	10	115	240
<u>Natural Gas</u>					
SCFM	375	300	-	-	675
<u>Refrigeration</u>					
0°F., tons				20.3	20.3
-20°F., tons	2,300	-	-	-	2,300
-50°F., tons	515	-	-	-	515
-100°F., tons	175	-	-	-	175
<u>Power, 3-Phase</u>					
440V, KW	2,620	820	40	30	3,510
<u>Nitrogen</u>					
SCFM	5	5	2	2	14
<u>Vacuum</u>					
25" Hg., CFM	20	20	5	35	80
18" mm. Hg. Abs.				20	20
1" mm. Hg. Abs.				20	20
<u>Compressed Air</u>					
60 psig, -20°F. dewpoint, SCFM	100	80	25	40	245
60 psig, 20°F. dewpoint, SCFM	10	10	10	10	40

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80 to 100 gpm to the scrubber. The scrubber off-gas passes through a gas-fired flare.

For vented gases from the pyrolysis plant and crystallization plant, only a flare is required since no acid gases are present. A liquid seal of inert liquid will be used to maintain nitrogen in the manifold system as used with the caustic scrubber.

B. Solids

The solids waste in the plant consists of polymeric boron hydrides and boric acid. Caustic solution from a portable 500 gallon tank can be used to clean out solids from fouled equipment.

Solids which are found in dismantling equipment which has not been removed by a caustic wash are inerted by decontaminating the equipment with 2.5 per cent ammonia solution. The ammonia solution will be available in the plant in several loosely-covered 55 gallon drums and used for personnel decontamination, equipment decontamination, and for washing down process liquor spills. A portable 500 gallon ammonia solution tank will also be available for equipment and area decontamination.

C. Tars

Tars formed in alkylation will be dissolved or diluted with kerosene and incinerated.

COSTS

Plant capital costs for an integrated high energy fuels plant with a nominal 9.5 ton/day capacity have been estimated at \$41,500,000. This provides a versatile plant capable of producing pentaborane, decaborane or HEF-3 starting with borax as the boron raw material source. Table 7 presents the cost and capacities for the various process plants and the cost of auxiliary plants and service facilities.

Table 8 gives a detailed, self-explanatory estimation of the manufacturing cost for producing solely pentaborane. This cost, \$1.45 per pound of pentaborane, is also typical of the operating cost for decaborane and HEF-3 since the differences between these costs is within the accuracy of the estimate. A 10 per cent makeup for both the recycle chlorine (from Step I HCl) and recycle hydrogen (from Step II offgas) is assumed. No insurance, taxes, or depreciation are included.

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TABLE 7

Estimated Capital Cost
9.5 tons/Day
AFN Boron High Energy Fuels Plant

	<u>Capacity</u> <u>Tons/Day</u>	<u>Cost</u>
<u>A. Process Plants</u>		
1. Boric Acid	64	\$ 1,500,000
2. Boric Oxide	36	1,250,000
3. Chlorine	100	2,500,000
4. Ethyl Chloride	5.4	150,000
5. Boron Trichloride	107	6,100,000
6. Diborane	11.9	9,800,000
7. Pentaborane-Decaborane	9.5 - 9.2	4,000,000
8. HEF-3	9.4	<u>4,000,000</u>
Subtotal		\$ 29,300,000
<u>B. Auxiliary Plants</u>		
1. Hydrogen	4.4	\$ 1,250,000
2. Nitrogen		1,710,000
3. Fire Protection, Water Supply, Waste Disposal		900,000
4. Cooling Tower		150,000
5. Powerhouse		<u>1,450,000</u>
Subtotal		\$ 5,460,000
<u>C. Service Facilities</u>		
1. Administration Building		\$ 450,000
2. Control Laboratory		900,000
3. Locker Room		100,000
4. Maintenance Building		400,000
5. Warehouse Buildings		200,000
6. Electrical Distribution and Switchgear		750,000
7. Roads and Fencing		<u>160,000</u>
Subtotal		\$ 2,960,000
Grand Subtotal		\$ 37,720,000
Contingency		<u>3,780,000</u>
Grand Total		<u>\$ 41,500,000</u>

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TABLE 8

Estimated Operating Costs
9.5 Tons/Day Pentaborane

Total Plant Cost \$36,500,000
Operating Days/Year 330

Raw Materials

	<u>lb./lb. B₅H₉</u>	<u>\$/lb.</u>	<u>\$/lb. B₅H₉</u>
Borax	10.6	0.022	0.233
Sulfuric Acid	2.8	0.010	0.028
Coke	1.9	0.018	0.034
Natural Gas	0.017 (MCF)	0.51 (MCF)	0.009
Chlorine	1.06	0.03	0.032
Cyclohexane	0.17	0.057	<u>0.010</u>
		Subtotal	<u>0.346</u>

Utilities

Electricity	15 (KWH)	0.009/(KWH)	0.135
Steam	0.2 (M lb.)	0.65/M lb.	0.130
Water	0.05 (M gal.)	0.07 (M gal.)	0.004
Natural Gas	0.04 (MCF)	0.51 (MCF)	<u>0.020</u>
		Subtotal	<u>0.289</u>

Labor & Misc.

Operating Labor	$\frac{(32 \text{ men})(\$3/\text{hr})(24 \text{ hr/da})(365 \text{ da/yr})}{(330 \text{ da/yr})(19000 \text{ lb/da})}$	0.134
Control Lab Labor	$\frac{(5 \text{ men})(\$3/\text{hr})(24 \text{ hr/da})(365 \text{ da/yr})}{(330 \text{ da/yr})(19000 \text{ lb/da})}$	0.021
Supervision	25% (0.134 + 0.021)	0.039
Operating Supplies	$\frac{0.5\% (36,500,000)}{(330 \text{ da/yr})(19000 \text{ lb/da})}$	0.029
Maintenance	$\frac{5\% (36,500,000)}{(330 \text{ da/yr})(19000 \text{ lb/da})}$	0.291
Overhead	100% (0.134 + 0.021 + 1/2 · 0.291)	<u>0.301</u>
	Subtotal	<u>0.815</u>
	Total Manufacturing Cost	<u>\$ 1.450</u>

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APPENDIX A

Equipment Design Summary

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APPENDIX A

Equipment Design Summary Diborane Production (Step I)

Item	Name	Size	Type	Material of Construction
<u>Reactors</u>				
1F-1	Reactor Preheater	6" dia. x 125'	Helical Coil	1/16" silver lined
1F-2	Reactor	6" dia. x 105'	Helical Coil packed/Ag screen	Type 310 SS as above
<u>Columns and Vessels</u>				
1C-1	Primary Absorber	48 trays, 5.5' dia. x 108'	Bubble Cap	C-Steel
1C-2	Secondary Absorber	32 trays, 4' dia. x 76'	Bubble Cap	Type 304 SS
1C-3	DCB Prefractionator	30 trays, 12' dia. x 88'	Bubble Cap	C-Steel
1C-4	Disproportionator	30 trays, 2.5' dia. x 70'	Bubble Cap	Upper Sec. Type 304 SS Lower Sec. C-Steel
1C-5	HCl Fractionator	26 trays, 8' dia. x 65'	Bubble Cap	C-Steel
1C-7,A,B,C,D	Diborane Storage Tank	270 gal.	Refrigerated w/internal coils	Type 304 SS
1C-8	Diborane Absorber	20 trays, 1.5' dia. x 48'	Bubble Cap	Type 304 SS
1C-9, A,B	HCl Storage Tanks	12,500 gal.	Refrigerated w/internal coils	Type 304 SS

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Equipment Design Summary
Diborane Production (Step I) (Cont.)

Item	Name	Size	Type	Material of Construction
<u>Heat Exchangers</u>				
1E-1	Reactor Feed Effluent Exchanger	31.6 MM B.t.u./hr.	Double Pipe	Shell and tube to be type 310 SS. Tube (both sides) and shell (inside) to be 1/16" silver lined.
1E-2	BCl ₃ Vaporizer	10.1 MM B.t.u./hr.	S & T	Type 304 tubes, C-St Shell
1E-3	Primary Absorber Trim Cooler	4.3 MM B.t.u./hr.	S & T	Carbon Steel
1E-4	Reactor Effluent Cooler	13.6 MM B.t.u./hr.	S & T	Carbon Steel
1E-5	Disproportionator Feed Chiller	2.0 MM B.t.u./hr.	S & T	Carbon Steel
1E-6	Recycle Hydrogen Cooler	1.1 MM B.t.u./hr.	S & T	Carbon Steel
1E-7	Disproportionator Feed Cooler	4.2 MM B.t.u./hr.	S & T	Carbon Steel
1E-8	Prefractionator Feed Primary Absorbent Exchanger	5.8 MM B.t.u./hr.	S & T	Carbon Steel
1E-9	Secondary Absorber Feed O.H. Exchanger	3.6 MM B.t.u./hr.	S & T	Carbon Steel
1E-10	Secondary Absorber Feed Chiller	10.5 MM B.t.u./hr.	S & T	Carbon Steel
1E-11	Secondary Absorber Feed IM "	2.0 MM B.t.u./hr.	S & T	Type 304 SS
1E-12	Secondary Absorbent HCl Fract. Feed Exchanger	43.5 MM B.t.u./hr.	S & T Long Baff.	Carbon Steel
1E-13	Secondary Absorbent Chiller	2.9 MM B.t.u./hr.	S & T	Type 304 SS
1E-14	Prefract. O.H. Condenser	67.9 MM B.t.u./hr.	S & T	Carbon Steel
1E-15	Prefract. Reboiler	78.2 MM B.t.u./hr.	S & T Thermo-siphon	Carbon Steel
1E-16	Disproportionator Reboiler	3.0 MM B.t.u./hr.	As above	Carbon Steel
1E-17	Disproportionator O.H. Cond.	2.1 MM B.t.u./hr.	S & T	Type 304 SS

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Equipment Design Summary
Diborane Production (Step I) (Cont.)

Item	Name	Size	Type	Material of Construction
<u>Heat Exchangers (cont.)</u>				
1E-18	HCl Fract. Reboiler	19 MM B.t.u./hr.	S & T Thermo-siphon	Carbon Steel
1E-19	HCl Fract. O.H. Cond.	9.2 MM B.t.u./hr.	S & T	Carbon Steel
1E-20	DB Absorbent Feed Bottoms Exchanger	1.2 MM B.t.u./hr.	S & T	Carbon Steel
1E-21	DB Absorbent Chiller	0.4 MM B.t.u./hr.	S & T	Type 304 SS
<u>Pumps and Compressors</u>				
1G-1 A,B	Recycle BCl ₃ Pump	185 gpm	Centrif.	Carbon Steel
1G-2 A,B	Secondary Absorber Bottoms Pump	1170 gpm	Centrif.	Type 304 SS
1G-3 A,B	Prefract. Bottoms Pump	1000 gpm	Centrif.	Carbon Steel
1G-4 A,B	Prefract. Reflux Pump	1500 gpm	Centrif.	Carbon Steel
1G-5 A,B	Disproportionator Bottoms Pump	100 gpm	Centrif.	Carbon Steel
1G-6 A,B	B ₂ H ₆ Product & Reflux Pump	54 gpm	Centrif. Can Pump	Type 304 SS
1G-7 A,B	HCl Product & Reflux Pump	120 gpm	As above	Type 304 SS
1G-8 A,B	DB Absorber Bottoms	88 gpm	Centrif.	Type 304 SS
1K-1 A,B	H ₂ Recycle Compressor	44,100 SCFM	Centrif.	Carbon Steel
1K-2 A,B	H ₂ Make-Up Compressor	2,200 SCFM	Centrif.	Carbon Steel

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Equipment Design Summary
Diborane Pyrolysis (Step II)

Item	Name	Size	Type	Material of Construction
<u>Reactor</u>				
2R-1	Pyrolysis Reactor	510 cu. ft. 11 mm B.t.u./hr.	Individually jacketed tubes	Carbon Steel
<u>Columns and Vessels</u>				
2C-1	B ₂ Absorber	40 trays, 4' dia. x 90'	Bubble Cap	Carbon Steel
2C-2	Absorber-Stripper	30 trays, 4' dia. x 70'	Bubble Cap	Carbon Steel
2C-3	B ₂ Stripper No. 1	50 trays, 4' dia. x 110'	Bubble Cap	Carbon Steel
2C-4	B ₂ Stripper No. 2	50 trays, 4' dia. x 110'	Bubble Cap	Carbon Steel
2C-5	B ₅ Distillation Column	20 trays, 3' dia. x 50'	Bubble Cap	Carbon Steel
2C-6	C ₆ Distillation Column	9 trays, 8' dia. x 34'	Bubble Cap	Carbon Steel
2C-7	B ₅ Conc. Column	40 trays, 2' dia. x 90'	Bubble Cap	Carbon Steel
2C-8	B ₅ Prod. Col.	30 trays, 1' dia. x 70'	3/4 Intalox Saddles	Carbon Steel
2PH-1	Reactor Phase Separator	4000 gal.		Carbon Steel
2PH-3	Gas Recycle Comp. K.O. Pot	200 gal.		Carbon Steel
2PH-4	Gas Recycle Comp. K.O. Pot	200 gal.		Carbon Steel
2C-9	Cyclohexane Make-Up Tank	500 gal.		Carbon Steel
2C-10 A-F	Pentaborane Storage	6 5000-gal.		Carbon Steel

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Equipment Design Summary
Diborene Pyrolysis (Step II) (Cont.)

Item	Name	Size	Type	Material of Construction
<u>Heat Exchangers</u>				
2E-1	Recycle Gas Preheater	7 mm B.t.u./hr.	S & T	Carbon Steel
2E-2	Quench Cooler	18 mm. B.t.u./hr.	S & T	Carbon Steel
2E-3	C ₆ H ₁₂ Make-Up Feed Cooler	1 m B.t.u./hr.	S & T	Carbon Steel
2E-4	Recycle Absorbent Cooler	920 m B.t.u./hr.	S & T	Carbon Steel
2E-5	Stripper-Absorber Liquid Effluent Interchanger	920 m B.t.u./hr.	S & T	Carbon Steel
2E-6	Stripper Liquid Heater	920 m B.t.u./hr.	S & T	Carbon Steel
2E-7	B ₂ Stripper No. 1 Liq. Heat Exchanger	450 m B.t.u./hr.	S & T	Carbon Steel
2E-8	B ₂ Stripper No. 2 Liq. Heat Exchanger	600 m B.t.u./hr.	S & T	Carbon Steel
2E-9	B ₅ Dist. Col. Reboiler	4.2 mm. B.t.u./hr.	S & T	Carbon Steel
2E-10	B ₅ Dist. Col. Condenser	3.0 mm B.t.u./hr.	S & T	Carbon Steel
2E-11	C ₆ Dist. Col. Reboiler	15 mm B.t.u./hr.	S & T	Carbon Steel
2E-12	C ₆ Dist. Col. Condenser	13 mm B.t.u./hr.	S & T	Carbon Steel
2E-13	B ₅ Conc. Col. Reboiler	780 m B.t.u./hr.	S & T	Carbon Steel
2E-14	B ₅ Conc. Col. Condenser	700 m B.t.u./hr.	S & T	Carbon Steel
2E-15	B ₅ Product Col. Reboiler	100 m B.t.u./hr.	S & T	Carbon Steel
2E-16	B ₅ Product Col. Condenser	100 m B.t.u./hr.	S & T	Carbon Steel
2E-17	Process Recycle Gas Precooler	120 m B.t.u./hr.	S & T	Carbon Steel
2E-18	Process Recycle Gas After-cooler	100 m B.t.u./hr.	S & T	Carbon Steel
2E-19	Absorber Off-Gas Heater	5 m B.t.u./hr.	S & T	Carbon Steel

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Equipment Design Summary
Diborane Pyrolysis (Step II) (Cont.)

Item	Name	Size	Type	Material of Construction
<u>Pumps and Compressors</u>				
2K-1 A,B,C	Reactor Recycle Gas Compressor	16,300 SCFM	Positive Displacement Rotary Screw Compressor	Carbon Steel
2K-2 A,B	Process Gas Recycle Compressor	600 SCFM	Centri. Compressor	Carbon Steel
2G-1 A,B,C	Reactor Liq. Recycle Pump	2000 gpm	Centrifugal	Carbon Steel
2G-2 A,B	K.O. Pot Liquor Pump	10 gpm	Turbine	Carbon Steel
2G-3 A,B	Pentaborane Product Pump	3 gpm	Diaphragm	Carbon Steel
2G-4 A,B	Absorbent Recycle Pump	220 gpm	Turbine	Carbon Steel
2G-5B	Absorber Feed Pump	75 gpm	Centrifugal	Carbon Steel
2G-6 A,B	B ₂ Stripper No. 2 Feed Pump	75 gpm	Centrifugal	Carbon Steel
2G-7 A,B	B ₂ Stripper No. 2 Effl. Pump	75 gpm	Centrifugal	Carbon Steel
2G-8 A,B	C ₆ Column Feed Pump	65 gpm	Centrifugal	Carbon Steel
2G-9 A,B	C ₆ Dist. Col. Pump	80 gpm	Centrifugal	Carbon Steel
2G-10 A,B	B ₅ Conc. Col. Bottoms Pump	76 gpm	Centrifugal	Carbon Steel
2G-11 A,B	C ₆ Fresh Feed Pump	0.66 gpm	Diaphragm	Carbon Steel
<u>Centrifuges</u>				
2V-1	Reactor Effluent Centrifuge	80 gpm 0.8 lb./min. solids	Cont. Press. Solids Discharge Split Bowl	Stainless Steel
2V-2A,B	B ₅ Dist. Col. Centrifuge	80 gpm 1.0 lb./min. solids	As Above	Stainless Steel

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Equipment Design Summary
Decaborane Crystallization (Step IIA)

Item	Name	Size	Type	Material of Construction
<u>Columns and Vessels</u>				
2C-11	B ₁₀ Holding Tank	15,000 gal.		Carbon Steel
2C-12	B ₁₀ Concentrator	400 gal.	Externally Heated Evaporator	Carbon Steel
2C-13	Concentrator Accumulator	100 gal.		Carbon Steel
2C-14	B ₁₀ Crystallizer	100 gal.	Externally Cooled, Agitated	Carbon Steel
2C-15	Mother Liquor Tank	50 gal.		Carbon Steel
2C-16	Product Storage Hopper	1250 cu.ft.	Internally Cooled Solids Storage	Type 304 SS
<u>Heat Exchangers</u>				
2E-9	Concentrator Reboiler	614 m B.t.u./hr.	S & T Thermosiphon	Type 304 SS
2E-10	Conc. O.H. Condenser	430 m B.t.u./hr.	S & T	Type 304 SS
2E-11	Crystallizer Cooler	120 m B.t.u./hr.	S & T	Type 304 SS
2E-12	Dryer Condenser	9 m B.t.u./hr.	S & T	Type 304 SS
2E-13	Dryer Jacket	16.4 m B.t.u./hr.	Vessel Jacket	Type 304 SS
2E-14	Dryer Gas Heater	700 B.t.u./hr.	Double Pipe Finned Tube	Type 304 SS
2E-15	Decaborane Prod. Cooler	44 m B.t.u./hr.	Internal Helical Coil	Type 304 SS
<u>Pumps</u>				
2G-12 A,B	B ₁₀ Transfer Pump	8 gpm	Centrif.	Type 304 SS
2G-13 A,B	Conc. Bottoms Pump	5 gpm	Centrif.	Type 304 SS
2G-14 A,B	C ₆ H ₁₂ Return Pump	5 gpm	Centrif.	Type 304 SS
2G-15 A,B	Crystall. Circulation Pump	7 gpm	Centrif.	Type 304 SS
2G-16 A,B	Filtrate Pump	3 gpm	Centrif.	Type 304 SS
2K-3 A,B	B ₁₀ Dryer Blower	10 MSCFH	Positive Displ. Lobe Blower	Type 304 SS
<u>Centrifuge</u>				
2V-3 A,B	B H Centrifuge	250 gal./hr. 765 lbs./hr. solids	Press. Cont. Disch.	Stainless Steel
<u>Dryer</u>				
2D-1	Decaborane Dryer	3' dia. x 30'	Rotary Steam Jacketed	Stainless Steel

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Equipment Design Summary
Decaborane Alkylation (Step III)

Item	Name	Size	Type	Material of Construction
<u>Reactor</u>				
3R-1	Alkylation Reactor	20 trays, 7.5' dia. x 50'	Bubble Cap	Carbon Steel
<u>Columns and Vessels</u>				
3C-1	Recycle Concentrator	400 gal.	Evaporator	Carbon Steel
3C-2	HCl Scrubber	2 trans. units 3' dia. x 6'	Packed Column 1" Intalox Saddles	Glass Lined Carbon Steel
3C-3	Alkylator Feed Tank	300 gal.		Carbon Steel
3C-4	C ₆ H ₁₂ Dist. Holding Tank	200 gal.		Carbon Steel
3C-5	C ₆ H ₁₂ Dist. Col.	4 trays, 3' dia. x 15'	Low Press. Drop High Vac. Mech.	Carbon Steel
3C-6	Recycle B ₁₀ Storage Tank	100 gal.		Carbon Steel
3C-7	Decaborane Dist. Col.	30 trays, 3' dia. x 70'	As above	Carbon Steel
3C-8	HEF Dist. Col.	40 trays, 2' dia. x 90'	As above	Type 304 SS
3C-9 A,B	HEF Storage Tanks	10,500 gal.		Carbon Steel
<u>Heat Exchangers</u>				
3E-1	Concentrator Reboiler	549 M B.t.u./hr.	S & T Thermo- siphon	Carbon Steel
3E-2	Conc. O.H. Condenser	375 M B.t.u./hr.	S & T	Carbon Steel
3E-3	Alkylator Feed Cooler	175 M B.t.u./hr.	S & T	Carbon Steel
3E-4	Alkylator Effl. Cooler	210 M B.t.u./hr.	S & T	Carbon Steel
3E-5	C ₆ H ₁₂ Dist. Reboiler	890 M B.t.u./hr.	S & T	Carbon Steel
3E-6	C ₆ H ₁₂ Condenser	812 M B.t.u./hr.	S & T	Carbon Steel
3E-7	Recycle C ₆ H ₁₂ Heater	60 M B.t.u./hr.	Integral Coil	Carbon Steel
3E-8	B ₁₀ H ₁₄ Dist. Reboiler	762 M B.t.u./hr.	S & T	Carbon Steel
3E-9	B ₁₀ Reflux Condenser	430 M B.t.u./hr.	Integral Finned Coil	Carbon Steel

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Equipment Design Summary
Decaborane Alkylation (Step III) (Cont.)

Item	Name	Size	Type	Material of Construction
<u>Heat Exchangers</u>				
3E-10 A,B	B ₁₀ H ₁₄ Condenser	280 M B.t.u./hr.	Integral Finned Coil	Carbon Steel
3E-11	HEF Dist. Reboiler	362 M B.t.u./hr.	S & T Thermosiphon	Type 304 SS
3E-12	HEF Reflux Condenser	362 M B.t.u./hr.	S & T	Type 304 SS
3E-13	Tars Cooler	500 M B.t.u./hr.	S & T	Carbon Steel
<u>Pumps and Compressors</u>				
3G-1 A,B	Recycle C ₆ H ₁₂ Pump	7 gpm	Centrif.	Carbon Steel
3G-2 A,B	Alkylator Bottoms Pump	10 gpm	Centrif.	Carbon Steel
3G-3 A,B	Deca Dist. Feed Pump	2 gpm	Centrif.	Carbon Steel
3G-4 A,B	Centrifuge Wash Liq. Pump	8 gpm	Centrif.	Carbon Steel
3G-5 A,B	Condenser Flush Pump	8 gpm	Centrif.	Carbon Steel
3G-6 A,B	HEF Dist. Feed Pump	2 gpm	Centrif.	Carbon Steel
3G-7 A,B	Tars Pump	0.5 gpm	Centrif.	Carbon Steel
3G-8 A,B	HEF Product Pump	3 gpm	Centrif.	Carbon Steel
<u>Centrifuges</u>				
3V-1 A,B	AlCl ₃ Centrifuge	9 gpm feed 350 lbs./hr. solids	Cont. Press., Solids Disch., Split Bowl	Stainless Steel

Note: Where two or more pieces of equipment are listed, (i.e., 2K-1A,B,C), one spare is indicated.

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APPENDIX B

Physical Properties of Process Chemicals

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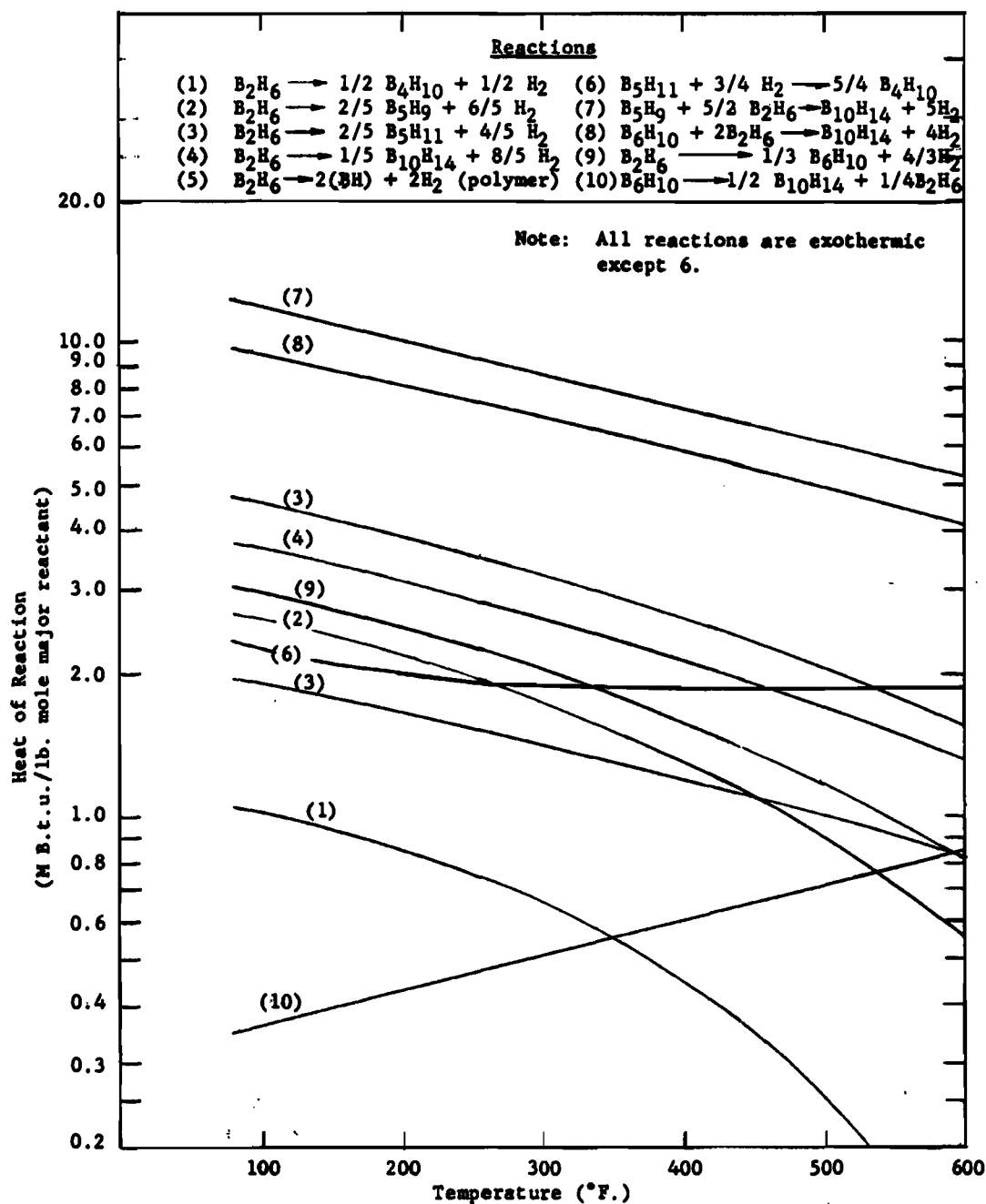
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Figure B-1. Diborane Pyrolysis - Estimated Heats of Reaction

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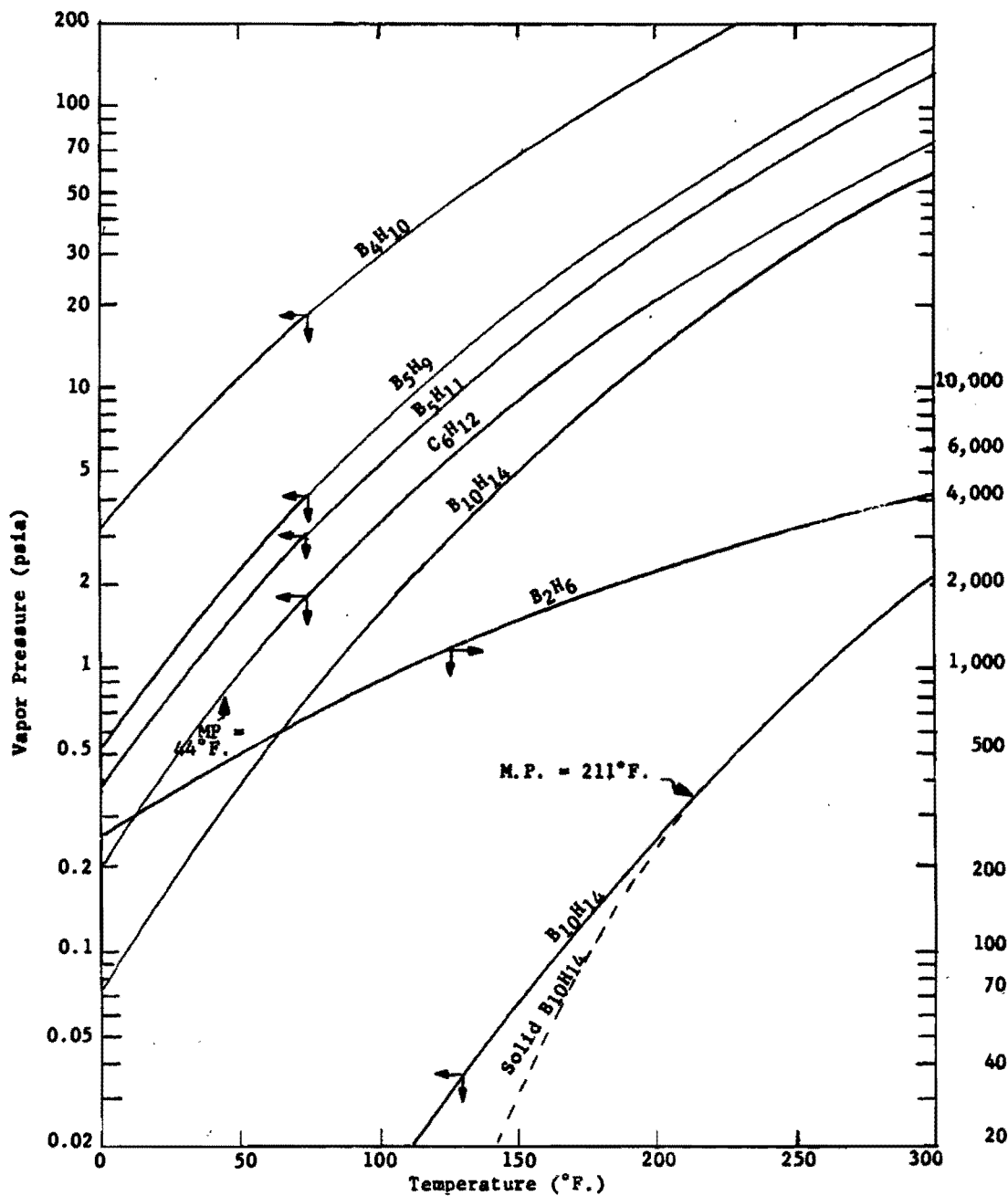
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Figure B-2. Vapor Pressure vs. Temperature



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B-2

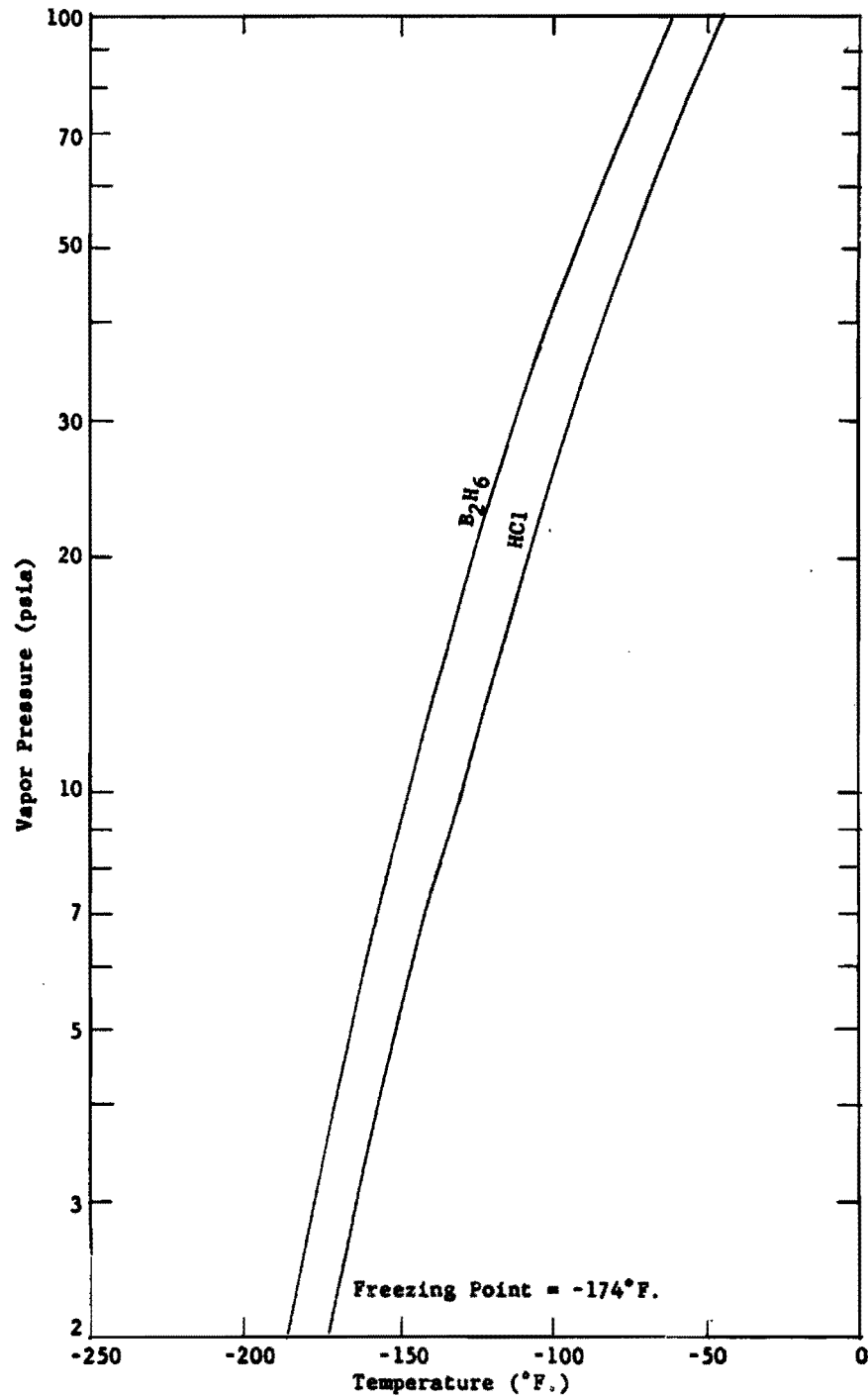
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June 1962

Figure B-3. Vapor Pressure vs. Temperature



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B-3

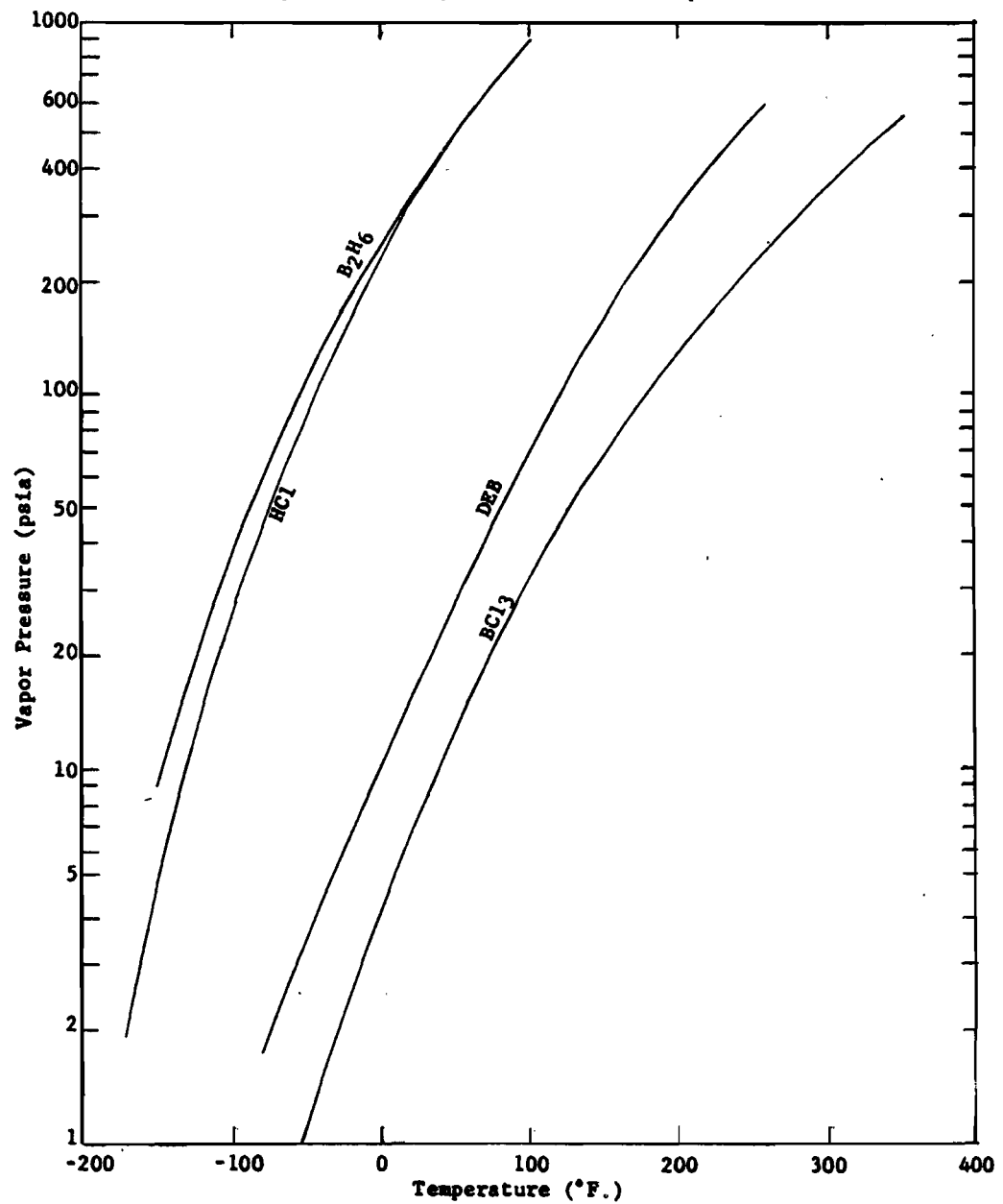
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Figure B-4. Vapor Pressure vs. Temperature



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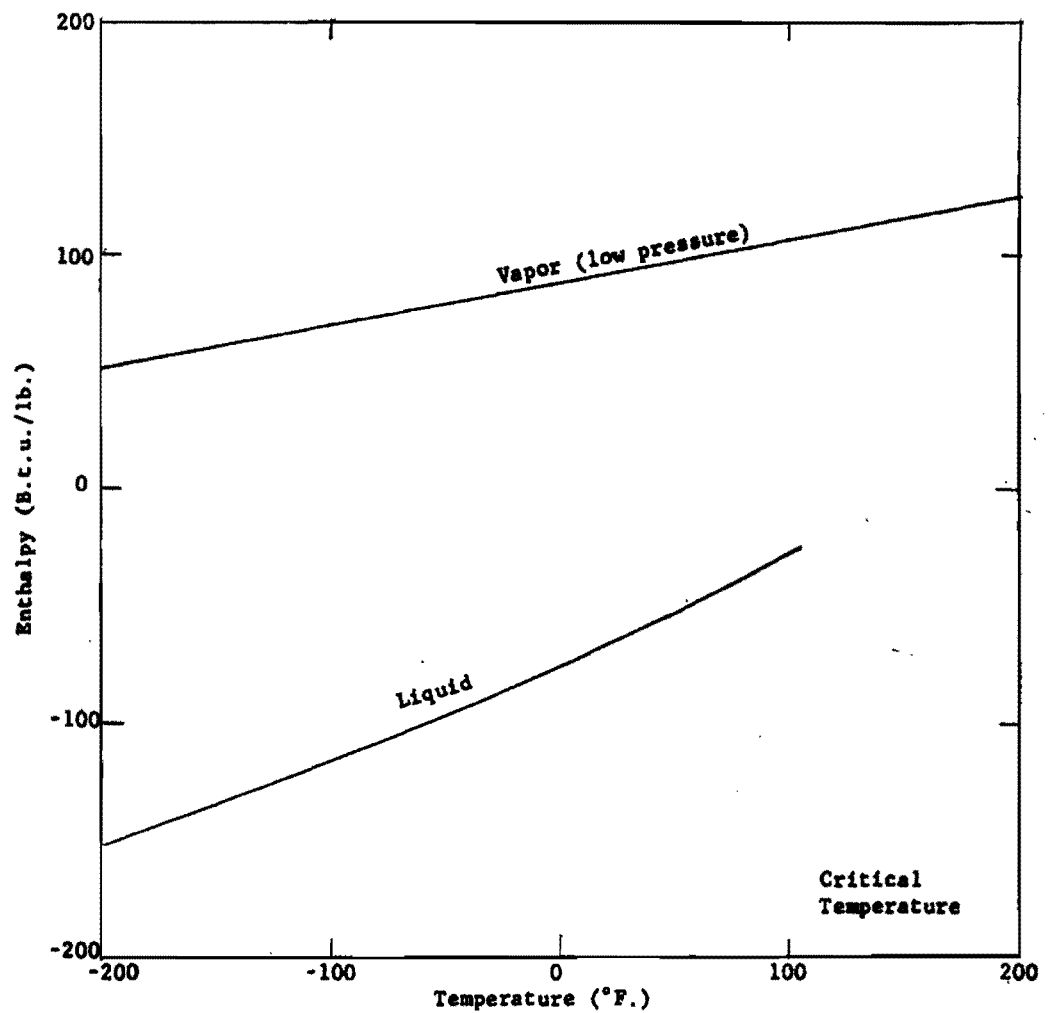


Figure B-5. Enthalpy of HCl (low range)

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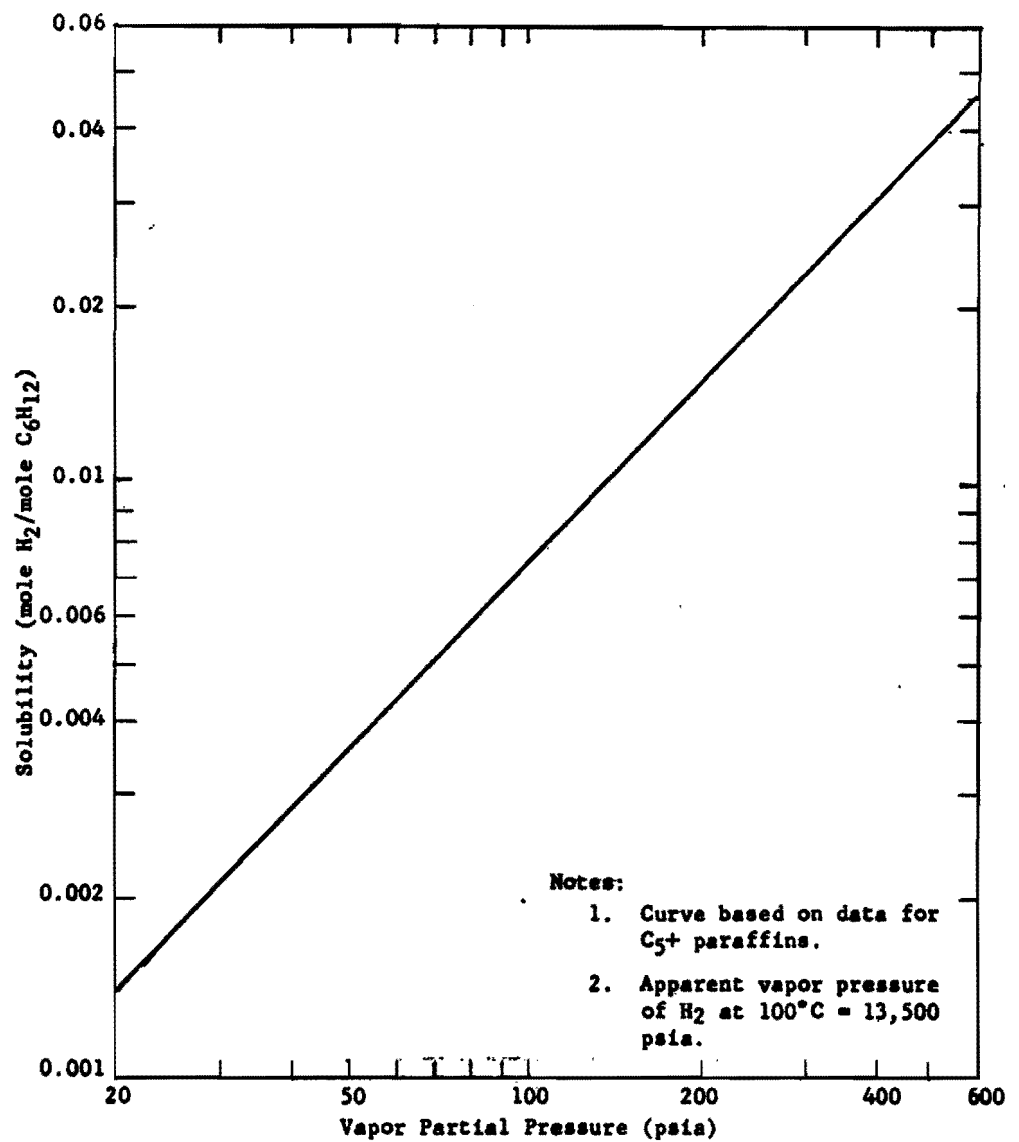


Figure B-6. Solubility of Hydrogen in Cyclohexane (100°C)

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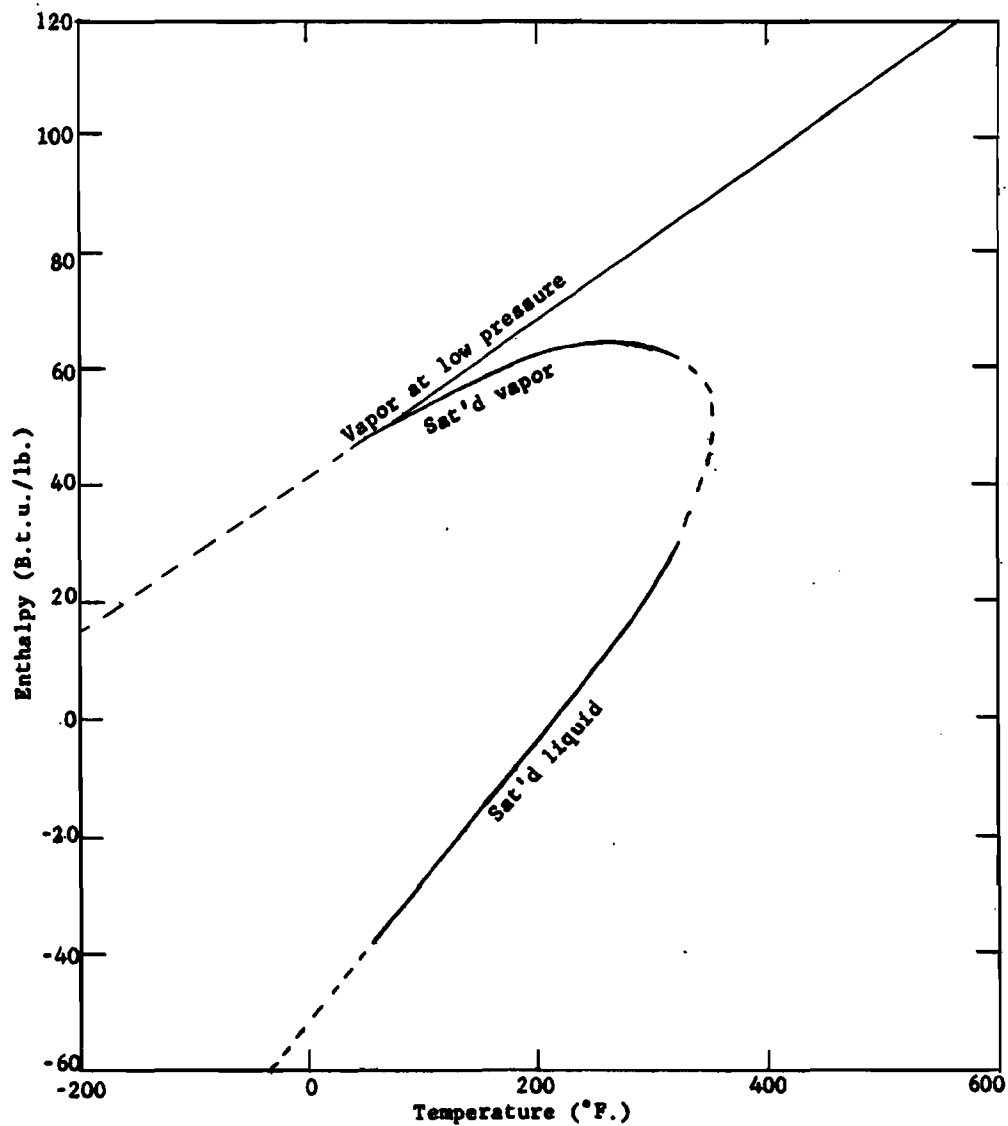


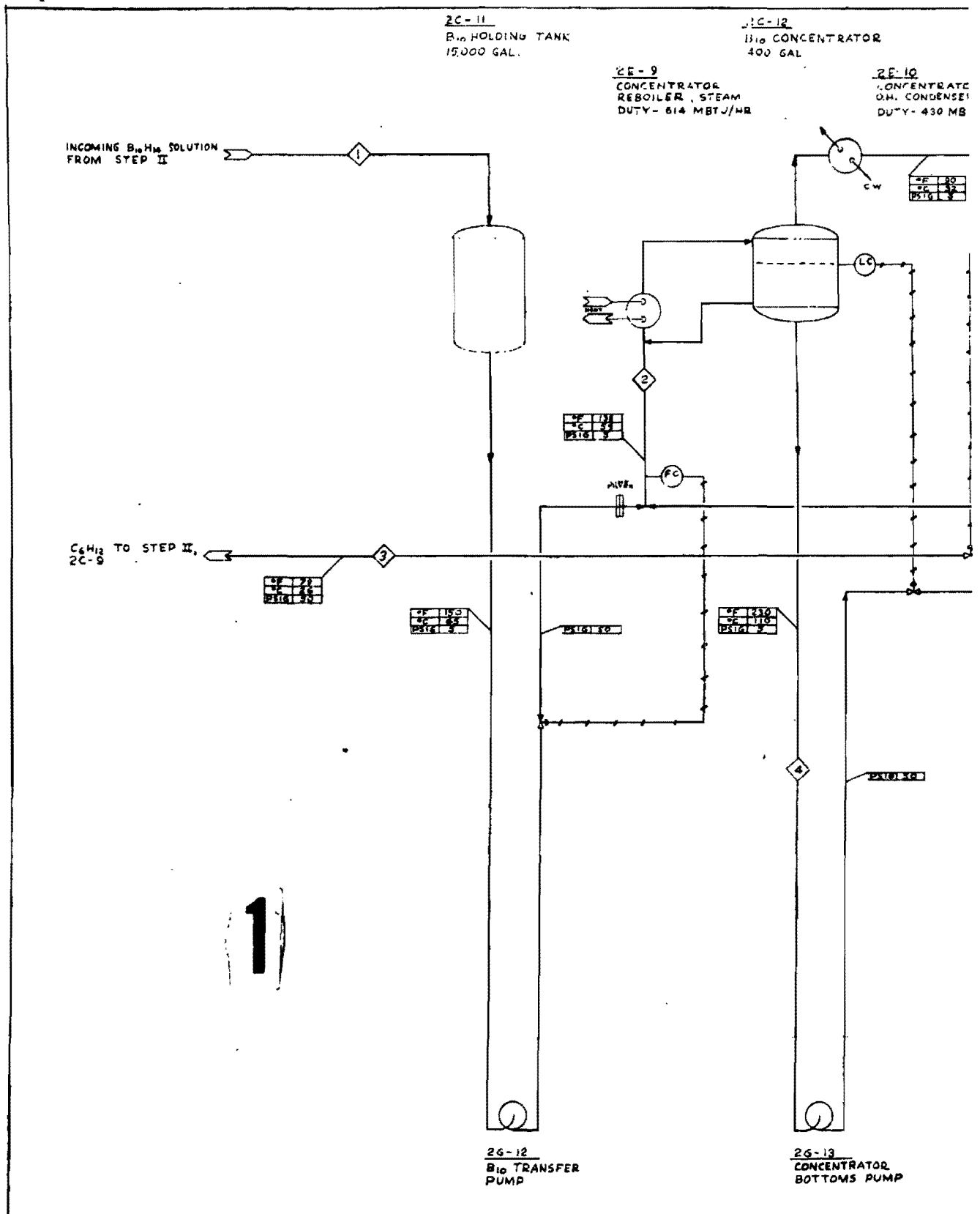
Figure B-7. Enthalpy of BCl₃

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DEPARTMENT OF THE AIR FORCE
HEADQUARTERS 88TH AIR BASE WING (AFMC)
WRIGHT-PATTERSON AIR FORCE BASE OHIO

16 Feb 2012

88 CS/SCOKIF (FOIA)
3810 Communications Blvd
Wright-Patterson AFB OH 45433-7802

Defense Technical Information Center
Attn: Ms. Kelly Akers (DTIC-R)
8725 John J. Kingman Rd, Suite 0944
Ft Belvoir VA 22060-6218

Dear Ms. Akers

This concerns Technical Report AD-337608, Preliminary Design for Large Scale Borane Plant." This record was previously "UNCLASSIFIED / LIMITED."

Subsequent to WPAFB FOIA Control Number 2012-01839-F, this record has been cleared for public release by Air Force Research Lab Propulsion Directorate Senior Chemical Engineer and Acting Branch Chief on 13 February 2012. Therefore, record is now fully releasable to the public. Attached on CD copy of it which has been remarked accordingly.

If you have any questions, please contact me at (937) 522-3091 or DSN 672-3091 or Lynn.kane@wpafb.af.mil.

Sincerely,

A handwritten signature in blue ink, appearing to read "L Kane", is written over the typed name.

LYNN KANE
Freedom of Information Act Analyst
Base Information Management Section
Knowledge Operations

3 Attachments

1. FOIA Request # 2012-01839-F
2. Copy of AFMC Form 559
3. CD with responsive records, remarked